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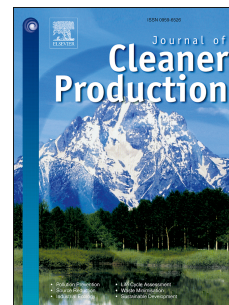
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# Journal Pre-proof

Utilisation of waste marble dust for improved durability and cost efficiency of pozzolanic concrete

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Conceptualization: Ideas; formulation or evolution of overarching research goals and aims

Took part in the methodology.

Took part in the supervision of the work.

Took part in the preparation of the manuscript.

Took part in the correction of the manuscript.

2. Ali Hamza:

Conducting part of the experimental work (Mechanical Properties)

3. Shahram Derogar:

Conducting part of the experimental work (Durability)

Preparation, creation and/or presentation of the published work, specifically writing the initial draft

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## **Utilisation of waste marble dust for improved durability and cost efficiency of pozzolanic concrete**

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**Abstract**

This study demonstrates that the incorporation of waste marble dust to pozzolanic concrete improves the long-term mechanical properties and durability characteristics. A comprehensive study utilising specimens containing a cement and silica fume binder were manufactured with incremental levels of marble dust fine aggregate. Important physical properties including compressive strength, water penetration depth, porosity, resistance to sulphate attack and resistance to freeze/thaw cycling were evaluated over a period of 1 year. Microstructural development attributed to cement hydration and pozzolanic reaction was imaged at 28 days and 1 year using scanning electron microscopy. The inclusion of marble dust greatly improved the salt crystallization and freeze and thaw resistance of the concrete over the long-term with only a small decrease in compressive strength observed. Importantly this highlights the beneficial properties of marble dust on durability. Additional advantages were shown through cost efficiency analysis which revealed that utilisation of marble dust and silica fume in concrete can reduce the embodied CO<sub>2</sub> emissions improving the economic credentials and environmental impact. Marble dust not only improves the physical characteristics but also provides an environmentally friendly route for waste disposal and creation of a more sustainable concrete.

**Keywords**

Material Properties, Scanning Electron Microscopy, Waste Reduction, Cost Efficiency

## 1. Introduction

Concrete has been extensively used in construction worldwide for more than 200 years (Guatam et al., 2014). Portland cement, required for the essential adherence in concrete however, results in a significant amount of carbon dioxide (CO<sub>2</sub>) release and other greenhouse gases (GHGs) (Malhotra, 2004). Besides the fact that it is one of the most energy intensive industries around the globe, cement manufacturing is threatening the environment and human health progressively (Aruntas et al., 2010; Utlu et al., 2006). Turkey alone produced approximately 72.5 million tons of concrete in 2018 (Arel, 2016a). Taking this into account, 1 ton of cement necessitates 1.5 tons of raw materials, 6 GJ of fuel and releases 0.94 tons of CO<sub>2</sub> to the atmosphere (Arel, 2016a). Nearly 68.15 million tons of CO<sub>2</sub> is released to the atmosphere in 2018 from the cement manufacturing practices in Turkey alone.

Every year, approximately 1.6 billion tons of cement and 10 billion tons of fine and coarse aggregates are consumed for concrete manufacturing practices globally (Mehta, 2001). 7 percent of the global carbon dioxide (CO<sub>2</sub>) emissions results from the annual cement production (Mehta, 2001). However, cement production is not the only practice contributing the carbon dioxide emissions for concrete making. Operating aggregates, such as extracting, processing, and carriage requires substantial amounts of energy and consequently produces enormous amounts of GHGs (Arel, 2016b). GHGs emissions, result from aggregate production, involve explosions for the blasting of rocks and stones, fuel oil used for transportation, loading and hauling, along with the electricity required for the crushing processes for aggregate production at a quarry (Sanal, 2018).

Concrete production that involves cement manufacturing and aggregate constitution is becoming an alarming concern of construction practice. Curtailing the industrial processes and enhancing the cost efficiency as well as decreasing the environmental pollution are imperative measures towards the sustainable development of construction industries for the prosperity of the earth and societal development. Incorporation of industrial wastes in concrete contributes towards the development of economical and environmentally friendly construction materials. Industrial by-products have been extensively used to partially replace materials to manufacture

concrete and similar construction materials. (Levy and Helene, 2004; Azevedo et al., 2018; Li et al., 2014 and Li et al., 2016). Marble dust, produced as a result of cutting in mines, has been widely used in concrete production within these materials.

A proper estimate regarding the amount of marble waste produced in Turkey cannot be achieved as no registry exists with respect to its generation. However, it is stated in Alyamac and Ince (2009) that 40 percent of world's marble reserves is found in Turkey. It is also reported<sup>3</sup> that marble reserves of Turkey are nearly  $3.872 \times 10^6 \text{ m}^3$  and in Afyon city alone, approximately  $125 \times 10^3 \text{ t/year}$  of marble is produced. Studies in the literature stated that during its production, approximately 20 to 25% of marble powder is being produced in the form of slurry (Haggar, 2007; Pareek, 2007). It is also stated in Singh et al. (2017) that 30 percent of marble is turned into waste because of some irregularities in form or smaller in size during processing. Reported figures demonstrated the significance of the amount of marble waste generation which causes severe environmental pollution (Sudarshan and Vyas, 2016). The disposal routes of these wastes could also be complex and could cause more harm for the surrounding areas and underground water reserves (Corinaldesi et al., 2010).

There are studies in the literature stating the use of marble waste as a replacement material to cement for concrete production (Uysal and Yilmaz, 2011; Alibdo et al., 2014; Belaisi et al., 2012; Rodrigues et al., 2015; Gesoglu et al., 2012; Gencel et al., 2012). For instance, Rodrigues et al., (2015) studies the 5, 10 and 20% marble dust incorporation as a replacement material for cement in concrete. Rodrigues et al. (2015) reported that compressive strength of concrete was not negatively affected by up to 10% marble dust substitution however, compressive strength of concrete was reduced by 25% when the substitution of marble dust was increased to 20%. A 13.46% decrease in the compressive strength of concrete was also reported when 20% marble dust is used as a cement replacement material (Gesoglu et al., 2012). A further study reported decreases in compressive strength of 91%, 86% and 76% in concretes containing 20, 30 and 40% marble dust, respectively, as a cement replacement material (Gencel et al., 2012). It is reported in a previous study that when more than 10% marble dust is used as a cement replacement material, capillary structure of concrete increases resulting in a substantial

increase in the pore structure of these materials and hence a significant decrease in the mechanical properties of concrete (Sanal, 2018). Although it is generally reported that the increase in the replacement levels of marble dust, used as cement replacement, results in adverse effects on the mechanical properties of concrete, distinctive results reported by individual researchers are regarded to be caused by the dissimilar tricalcium aluminate ( $C_3A$ ) contents of cement utilised in these investigations (Gesoglu et al., 2012). The reactions between the tricalcium aluminate ( $C_3A$ ) in cement and calcium carbonate ( $CaCO_3$ ) in marble dust results in a formation of calcium carboaluminate that accelerates both the hydration rate and the development of compressive strength of concrete (Arel, 2016b).

Studies in the literature that investigated the use of marble dust as a replacement material to sand in concrete, stated that marble dust acts as a filler material in the cement matrix and thus causes a considerable decrease in water absorption and porosity which results in a substantial increase in compressive strength (Gamerio et al., 2014; Talah et al., 2015; Belachia and Henhoub, 2011; Hameed and Sekar, 2009; Hebhoub et al., 2011; Omar et al., 2012). It is reported in Hameed and Sekar (2009) that compressive strength of concrete was nearly 10% higher at 28 days when a 50% replacement level of marble dust was substituted for sand. It is further shown in Hebhoub et al. (2011) that a 28 day compressive strength of concrete incorporating 25, 50 and 75% marble dust as a sand replacement, was increased by 22.2, 16.8 and 16.8 respectively. It has also been demonstrated in Omar et al. (2012) that the use of 15% marble dust as a sand replacement resulted in an increase in the elastic modulus of around 5% and noticeably increased the compressive strength. In view of the aforementioned studies, it can generally be concluded that an increase in the replacement level of marble dust, used as sand replacement, has a positive influence on the mechanical properties of concrete. However, it is also stated in studies (Hameed and Sekar, 2009; Hebhoub et al., 2011; Demirel, 2010) that an increase in the replacement level of marble dust, particularly from 50% onwards, could result in an apparent increase in porosity that also increases the capillary action and water absorption and therefore causes a considerable decrease in the compressive strength of concrete as a result of the non-homogenous dispersal of the marble dust within the cement matrix (Arel, 2016b).

In light of the above studies, marble dust is used as a sand replacement and silica fume, a pozzolanic substitution, is used as a cement replacement in concrete. Pozzolanic substitution can be used to compensate for any reduction in mechanical properties of concrete incorporating marble dust.

The aim of this paper is to investigate the possible role of marble dust used as a replacement to fine aggregates on the long-term mechanical properties and durability characteristics of pozzolanic concrete. The parameters investigated are the compressive strength, water penetration depth, porosity, resistance to sulfate attack, and resistance to freeze and thaw over the long-term. The microstructural analysis performed are supported with Scanning Electron Microscopy. Elemental composition of the raw materials were obtained from the X-ray fluorescence and crystalline phases are identified using X-ray diffraction. The cost-efficiency of concrete incorporating marble dust and silica fume were analysed and consequently the positive contributions to decrease the CO<sub>2</sub> emissions in order to improve the economic recovery and to reduce environmental pollution are addressed in the paper. The results reported in this paper are significant as they provide a comprehensive insight into the effects of marble dust particularly on the long-term mechanical properties and durability characteristics of pozzolanic concrete. Paper begins with an introduction addressing the sustainable aspects of concrete consumption along with the difficulties met for the waste management of marble dust. Paper then reports the materials used and mix design principles adopted for the formation of test specimens in Section 2. Experimental procedures that covers mechanical, physical and durability properties are reported in Section 3. Results and discussions of the incorporation of marble dust, used as a replacement to fine aggregates, on the long-term mechanical properties and durability characteristics of pozzolanic concrete are provided in Section 4 in detail. Paper is concluded in Section 5 with the emphasis that the use of such industrial wastes enhances the materials properties of concrete for the use in construction practise as well as decreases the CO<sub>2</sub> emissions significantly, that suggest alternatives routes for improving the local economic recovery and reducing environmental pollution.

## **2. Materials and Mix Design**

The experimental study includes cement, silica fume, marble dust, fine aggregates, coarse aggregates and water as the main constituents for concrete manufacture. Portland cement was obtained from Adana Cimento Ltd. and conforms to ASTM C150/C150M-16. The silica fume, used as a partial replacement material to cement, was procured by Cyprus Environmental Enterprises Ltd. (CEE) and conforms to ASTM C1240-15. Marble dust, used as a partial replacement to fine aggregates, were obtained from Izmer Ltd. The aggregates used in the concrete were obtained from local limestone rock reserves. Typical fine and coarse aggregate gradations were obtained in accordance to ASTM C128 (2007) and ASTM C127 (2007) respectively. Physical properties of cement, silica fume and marble dust are summarised in Table 1. A water to binder (w/b) ratio of 0.42 was applied to all the concrete mixes investigated. It must be noted however that a constant ratio of 0.42 was achieved by replacing the fine aggregate with the particle size of only 0.5 mm and less than 0.5mm in order to minimize the differences in the fineness of these raw materials. The control concrete specimen is denoted with C. Concrete specimens incorporating 20% silica fume as a cement replacement is then identified as C20SF. Concrete specimens incorporating 20% silica fume as cement replacement and 10% and 20% marble dust as fine aggregate replacements were then identified as C20SF+10MD and C20SF+20MD respectively. Three 150mm x 150mm x 150mm cube shaped concrete specimens were prepared for each experiment type at all specified measurement times. Following the completion of cast work, concrete specimens were caged with a moist duster for 24 hours and then were de-moulded and stored in the curing tank at a temperature of  $24 \pm 2.0^{\circ}\text{C}$  until the specified test dates. ACI 211 principles are followed to obtain the mix constituents of concrete in this study. The mix constituents of concrete specimens incorporating silica fume and marble dust are summarised in Table 2. Slump was maintained at a constant value of 100mm for all the concrete mixes described in this study and are in a good correlation with the Marsh Cone Flow values of cement grout incorporated waste marble dust and fly ash (Cinar et al., 2019).

### **3. Experimental Procedure**

#### **3.1 Compressive Strength**

Compressive strength tests were performed on concrete incorporating silica fume as a partial replacement to cement along with marble dust as a partial replacement for fine aggregates. Standard concrete cubes were tested using a compressive strength testing machine at 28 days, 3 months, 6 months, 9 months, 12 months and 36 months respectively. The loading rate was kept constant at 0.6 MPa/s. Average compressive strengths were then computed for all the results for each replacement level (BS EN 12390-3).

### **3.2 Water Penetration Depth**

Water penetration depth of standard concrete cubic samples incorporating silica fume, as a partial replacement to cement, and marble dust, as a partial replacement to fine aggregates, was carried out at 28 days, 3 months, 6 months, 9 months and 12 months respectively. A water pressure of  $500 \pm 50$  kPa was applied to the concrete cubic samples for 72 hours during the experiment. Following 72 hours of the water pressure exposure, samples were removed from the water permeability test equipment. The exposed faces of the specimen were wiped immediately to remove excess water. Using the split tensile strength test machine, the samples were then split into half, perpendicularly to the face on which the water pressure was applied. The maximum depth of water penetration under the test area was measured. Measurements were recorded to the nearest mm (BS EN 12390-8). The initial mass,  $m_i$ , of the concrete samples were measured following air-drying prior to the experiment. Following this the samples were subjected to 72 hours of water pressure, the final mass,  $m_f$ , of the samples was then obtained before they were split in half. The difference in mass of the samples before and after the experiment was calculated by subtracting  $m_i$  from  $m_f$ .

### **3.3 Resistance to sulphate attack**

Resistance to external sulphate attack of concrete samples was determined by measuring the volumetric change and compressive strength. A sulphate solution was prepared using 50 g of  $\text{Na}_2\text{SO}_4$  in 1000 g of water in accordance with ASTM C1012/C1012M-15. Concrete samples were cured under water for 28 days before being exposed to the sulphate solution for a further 28 days, 3 months, 6 months, 9 months and 12 months respectively. The change in length was determined by in accordance with ASTM C490/C490M-1. Average length change was reported

to the nearest 0.01% in millimetres for each concrete replacement sample. The samples were air-dried before and after placing into the sulphate solution prior to length measurements. Compressive strength tests were also performed to examine the resistance of the concrete samples to external sulphate attack.

### **3.4 Resistance against Freeze and Thaw**

The concrete samples resistance to repeated freezing and thawing cycles was determined based on the standard procedure described in ASTM C666/C666M-15 using the freeze and thaw chamber with controlled temperature and humidity. The temperature was fluctuated within the range -30°C to 30°C and relative humidity fluctuated within the range 75% to 85%. Concrete samples were placed in the freeze and thaw chamber following water curing for 28 days. The chamber was set to perform two continuous cycles of freeze and thaw each day. The resistance to freeze and thaw of concrete samples was then determined by conducting compressive strength tests at 28 days, 3 months, 6 months, 9 months and 12 months following exposure in the chamber. The air-dried mass of the samples, recorded prior to the freeze and thaw cycles, and mass after they were subjected to proposed number of cycles was measured and hence the mass loss of the concrete samples during freeze and thaw cycles could be calculated.

### **3.5 Porosity**

Porosity of the concrete samples was determined based on a standard procedure described in ASTM D4404-10 using Mercury-intrusion porosimetry. This requires the progressive intrusion of mercury into a porous structure under controlled pressure. As the mercury has a very high contact angle with the concrete it cannot penetrate through pores by capillary action alone and requires an external pressure. The pressure to force the mercury into the pores can be used to determine the pore diameter while the volume of mercury displaced provides the corresponding pore volume. The mercury intrusion porosimetry test was performed at 1 year.

### **3.6 Scanning Electron Microscopy**

Microstructural investigations were performed using the scanning electron microscope. A thin layer of gold/platinum was sputtered onto the surface to reduce charging under the electron



beam. The development and the ongoing process of cement hydration, pozzolanic reaction, as well as the influence of the presence of marble dust was determined by microstructural analysis. The analysis was carried out on the samples at 28 days and 1 year respectively.

### 3.7 X-ray Diffraction

X-ray diffraction was used to determine the crystalline phase composition of the silica fume and marble dust. Powder samples were analysed using a Bruker AXS D8 Advance X-ray Diffractometer equipped with a superspeed PSD:Vantec-1 detector and  $\text{CuK}_\alpha$  X-ray source. Data was obtained in the 2-theta range 10 to 70°, with a step size of 0.023° and time of 0.425 seconds per step. X-ray diffraction patterns were background corrected using AXS software (Bruker Corporation, Germany). Peak identification was carried out using Crystal Impact Match version 3.4.2 software using the International Centre for Diffraction Data (ICDD) PDF-2 database.

### 3.8 Cost Efficiency Factor

Cost efficiency factor (CEF) of concrete samples incorporating silica fume and marble dust was determined using Equation (1). In Eq. 1 cost efficiency factor (CEF) is the ratio of the compressive strength of concrete to the total material cost per  $\text{m}^3$  (Agarwal and Gulati, 2006). The local prices of constituents at the time of the study in North Cyprus are employed to pursue the cost estimates and are stated in dollars in this paper. \$0.12 per kg of cement, \$0.21 per kg of silica fume, \$14.86 per  $\text{m}^3$  of fine aggregates and \$13.85 per  $\text{m}^3$  of coarse aggregates are used to define the cost efficiency factor.

$$CEF = \frac{F_c}{C} \times 100 \quad (1)$$

Where  $F_c$  is the average compressive strength of concrete and C is the cost of materials per  $\text{m}^3$  of concrete.

## 4. Results and Discussion

### 4.1 Characterisation of raw materials

The chemical composition of the raw materials, obtained from X-ray fluorescence analysis, are summarized in Table 3. Silica fume contains 90% of silicon dioxide ( $\text{SiO}_2$ ) and therefore has a high pozzolanic activity. Marble dust, broadly described as a limestone powder in nature shows 60% calcium when analysed as the oxide. The particle size distribution of raw materials are also presented in Figure 1. The particle size distribution of marble dust shows a high fineness compared with fine aggregates. Figure 2(a-c) show SEM images of the cement, marble dust and silica fume in the as-received state. Figure 2(a) shows the cement consists of particles ranging in size from sub-micron up to approximately 20 microns. Spherical pulverised fuel ash (pfa) particles which are blended with the cement during production are clearly visible distributed evenly amongst the clinker particles. The marble dust is shown in Figure 2(b), where the particles show a range in size from sub-micron up to approximately 20 microns. Both these ranges are in broad agreement with the measured particle size distributions. Figure 2(c) shows a typical image of the silica fume particles, these range from nano-sized ( $<100\text{nm}$ ) up to approximately  $400\text{nm}$ . It is noteworthy that the particle size analysis indicated that particle sizes were up to  $400\text{nm}$ . This suggested that during the particle size analysis the particles were agglomerated together resulting in an unrepresentatively large particle size being reported. This is also a typical incidences of the use of high-volume pozzolan which is demonstrated in Shakiba et al. 2018. Chemical composition, particle size distribution as well as the microstructural properties of marble dust have common features with granite residue substituted in cement mortars by Azevedo et al. (2019) and kaolinitic clay substituted in hydrated lime mortars by Marvila et al. (2019b).

X-ray diffraction patterns for the Marble dust and Silica fume are presented in Figure 3. Two phases were identified in the pattern corresponding to marble dust. The first phase, a calcite containing magnesium, was identified by the presence of peaks at 2-theta values of  $23.3^\circ$ ,  $29.7^\circ$ ,  $31.7^\circ$ ,  $36.2^\circ$ ,  $39.6^\circ$ ,  $43.4^\circ$ ,  $47.4^\circ$ ,  $48.8^\circ$ ,  $56.8^\circ$ ,  $57.7^\circ$ ,  $61.1^\circ$  and  $64.9^\circ$ . The presence of magnesium within the calcite phase is corroborated by the XRF analysis where magnesium was identified. The second phase corresponded to  $\text{SiO}_2$  in the form of quartz, and was identified by peaks at 2-theta values of  $21.1^\circ$ ,  $26.9^\circ$ ,  $36.4^\circ$ ,  $50.4^\circ$  and  $60.2^\circ$ . A quantitative analysis carried out by the software 'Crystal Impact Match' estimated a quartz content of approximately

20%. The X-ray diffraction pattern corresponding to the silica fume shows a broad peak centred at a 2-theta value of  $22^\circ$ . This indicates that the silica fume has a highly amorphous structure and that no crystalline impurity phases were present within the material.

#### 4.2 Strength

Concrete specimens were prepared using 20% silica fume as a replacement material to cement in combination with marble dust as a replacement material for fine aggregates. Replacement levels of marble dust were 10 and 20%. Long-term compressive strength of concrete specimens, all cured under water, are shown in Figure 4. Results shown in Figure 4 indicate that the use of silica fume enhanced the compressive strength of the concrete particularly over long-term durations. This is solely attributed to the pozzolanic activity of silica fume which accelerates the formation of calcium-silicate-hydrate (C-S-H) gels in cement matrix. It is also shown in Figure 4 that an increase in the replacement level of marble dust results in a slight decrease in compressive strength of concrete. It must be noted however, that the strength values obtained at all combinations with marble dust are increasing with time and are much higher than the control concrete specimen at all times. Standard error, shown in Figure 4, does not exceed 3.3 MPa for all the test groups examined in the paper and fully complies with ASTM C94/C94M.

The microstructural images of the cement control specimens and specimens containing silica fume, marble dust, and silica fume and marble dust at ages of 28 days and 1 year are shown in Figures 5 (a-h). Figures 5(a) and (b) show the microstructure of the pure cement specimens. The microstructures of the cement specimens containing 20% silica fume at 28 days and 1 year are shown in Figures 5(c) and (d) respectively. At 28 days, substantially more silicate needles, attributed to the much higher silica content, are evident and attributed to the addition of silica fume. At 1 year the microstructure is more consolidated and resembles the control specimen more closely. Figures 5(e) and (f) show the 28 day and 1 year old specimens of the mix containing 20% marble dust. Marble dust particles are clearly shown in the 28 day specimen surrounded by the cement matrix. The C-S-H microstructure is more consolidated after 1 year compared to at 28 days. Figures 5(g) and (h) show the 28 day and 1 year old specimens of the

mix containing 20% silica fume and 20% marble dust. At 28 days a large amount of finely divided silicate crystals are observed on the surface of the specimen. After 1 year these structures are more consolidated and a more uniform structure consisting of small particles bound together with limited porosity is observed.

#### **4.3 Water Penetration Depth**

The durability of concrete and resistance to adverse external and internal conditions can be influenced through its permeability. However, in the study for practical purposes the water penetration depth method was employed to test the dense concrete specimens. The water penetration depth of concrete specimens incorporating silica fume and marble dust are shown in Figure 6. The results indicate that the use of silica fume resulted in a significant decrease in the water penetration depth of concrete. The results shown in Figure 5 also illustrate that the increase in the replacement level of marble dust led to an even more substantial decrease in the water penetration depth of the concrete. This behaviour is expected as the coarse particles of sand are partially replaced with marble dust leading to a decrease in free volume within the cement matrix and therefore resulting in a more impermeable concrete. The results shown in Figures 5(a) and (b) and Figures 5(g) and (h), respectively, indicate a decrease in the capillary pore volume of the concrete associated with the incorporation of silica fume and marble dust. This supports the denser microstructure observed in the specimens containing silica fume and marble dust compared to the control concrete specimen. The mass of absorbed water through the application of the pressurised water during the experiment is plotted in the secondary axis in Figure 6. Results indicate that the use of silica fume coupled with the use of marble dust results in a systematic decrease in water penetration depth as well as a systematic decrease in the mass of absorbed water during the experiment.

#### **4.4 Resistance to Sulphate Attack**

The resistance to sulphate attack of concrete incorporating silica fume as a cement replacement and marble dust as a fine aggregate replacement is investigated in this section. It was essential to attain significant hydration of concrete prior to sulphate attack exposure therefore, the samples were cured for 28 days prior to placement in the sulphate solution. The results shown

in Figure 7 indicate that the concrete control specimen generated the strength loss more than those with silica fume and marble dust. Increasing replacement levels of marble dust resulted in a systematic decrease in the strength loss of concrete. This suggests that incorporation of both silica fume and marble dust resulted in an increased resistance to sulphate attack of the concrete. This behaviour is partly attributed to the pozzolanic reaction between the silica fume and calcium di-hydroxide,  $\text{Ca(OH)}_2$ , resulting in the formation of additional C-S-H phases. In addition to the formation of a denser microstructure, the consumption of  $\text{Ca(OH)}_2$ , used as a reactant in the sulphate reaction, tended to reduce the extent of sulphate attack. The decrease in compressive strength loss of concrete was also attributed to the fine particle size of marble dust that led to an increasing density and hence impermeability of the concrete. It should be accentuated that ~37% decrease in the compressive strength loss of concrete containing 20% silica fume and 20% marble dust is obtained compared to concrete containing 20% silica fume alone. 37% decrease in the compressive strength loss of concrete containing 20% silica fume and 20% marble dust validates the significance feature of marble dust on the increased resistance of sulphate attack of silica fume concrete.

It is a known phenomenon that the formation of ettringite and gypsum during sulphate attack on concrete results in an increase in volume and hence results in expansion and subsequent cracking of the concrete (Tian and Lohen, 2000). Results shown in Figure 7 indicate that the concrete control specimen subjected to sulphate attack exhibited a much higher expansion compared to the specimen incorporating 20% silica fume and 20% marble dust.

SEM images of the cement control, cement and 20% silica fume and cement with 20% silica fume and 20% marble dust exposed to sulphate testing at 28 days and 1 year are shown in Figures 8 (a-f). Figures 8(a) and (b) show the microstructures of the cement control specimen exposed to the sulphate testing for 28 days and 1 year. The structures are well consolidated with limited porosity visible in each image. Figures 8(c) and (d) show the microstructures of the cement with 20% silica fume specimen exposed to the sulphate testing for 28 days and 1 year. Compared to the pure cement specimens, the effect of the silica fume is evident through the much larger quantity of high aspect ratio silicate needles observed. At 28 days a large number

of pfa particles can be seen, however at the age of 1 year these are less visible and have been enveloped by C-S-H phases. Figures 8(e) and (f) show the microstructures of the cement with 20% silica fume and 20% marble dust exposed to the sulphate testing for 28 days and 1 year. Both images show marble dust particles surrounded by the C-S-H matrix, these are more visible at 28 days. At 28 days the silicate needles are more evident compared to 1 year where a more compact and consolidated structure is observed.

#### **4.5 Resistance to Freeze and Thaw Cycling**

The resistance to freeze and thaw of concrete incorporating silica fume as a cement replacement and marble dust as a fine aggregate replacement was investigated. It is noteworthy that the concrete specimens were water cured for 28 days before they were placed into the freeze and thaw chamber. The results shown in Figure 9 demonstrate that concrete control specimens subjected to freeze and thaw cycles yielded a greater strength loss compared to the concrete specimens with silica fume and marble dust. The use of silica fume and marble dust enhanced the freeze and thaw resistance of concrete significantly. Results shown in Figure 9 also suggest that concrete control specimens yielded a greater mass loss compared to the concrete specimens incorporating silica fume and marble dust. These results are in a good agreement with the compressive strength loss of concrete. As the number of freeze and thaw cycles are increasing, the subsequent hydraulic pressure in the matrix system was increasing which resulted in a progressive development of micro-cracks and hence led to a mass loss of the concrete specimen. The formation of cracks and hence the mass loss of the concrete specimens obviously led to a greater loss of strength in those samples. It must be emphasised that compared to concrete containing 20% silica fume alone, 62% decrease in the compressive strength loss of concrete containing 20% silica fume and 20% marble dust is obtained. The significant decrease in the compressive strength loss demonstrated the substantial contribution of marble dust alone on the increased resistance of freezing and thawing of silica fume concrete.

SEM images of the cement control, cement with 20% silica fume and cement with 20% silica fume and 20% marble dust exposed to freeze and thaw testing at 28 days and 1 year are shown

in Figures 10 (a-f). Figures 10(a) and (b) show the microstructures of the cement control specimen exposed to the freeze and thaw testing for 28 days and 1 year. These specimens look similar to the control specimens shown in Figure 5(a) and (b), however cracking is evident within the structure. This is particularly apparent in Figure 10 (b) where a crack propagating in the roughly horizontal direction is shown in the top left hand side of the image. Figures 10(c) and (d) show the microstructures of the cement with 20% silica fume specimen exposed to the freeze and thaw testing for 28 days and 1 year. Compared to the control specimen cracking is less evident. This may be a consequence of the higher strength attributed to the presence of silica fume and the associated C-S-H phases formed. Figures 10(e) and (f) show the microstructures of the cement with 20% silica fume and 20% marble dust exposed to the freeze and thaw testing for 28 days and 1 year. Marble dust particles are observed in both images and also silicate needles associated with the formation of C-S-H. Interestingly the cracking observed in the control specimen is not evident suggesting that the combination of silica fume and marble dust additions help enhance the freeze thaw resistance.

It can be concluded that the microstructural images shown in Figures 10(c) and (d) indicate the increased resistance of the concrete to freeze and thaw is attributed to the formation of additional C-S-H crystals as a result of the pozzolanic reaction of between the silica and  $\text{Ca(OH)}_2$ . Figures 10(e) and (f) also shows that concrete specimens containing silica fume and marble dust had a greater reduction in the capillary pores that played a significant role in increasing the freeze and thaw resistance of the concrete. Degree of saturation is one of the major factors that affects the concrete freeze and thaw resistance. The results shown in Figure 6 confirmed that concrete specimens incorporating silica fume and marble dust have much lower water penetration depths and hence led to a lower permeability and therefore less water imbibition in wet conditions. Microstructural images shown in Figure 10(e) and (f) also support the development of a less permeable cement matrix with the incorporation of silica fume and marble dust and hence improved freeze and thaw resistance of concrete.

#### **4.6 Porosity**

Effects of using marble dust on the porosity of pozzolanic concrete under various cure conditions are investigated in this section. Concrete specimens incorporating 20 percent silica fume as a partial replacement to cement along with 10 and 20 percent marble dust as a partial replacement to fine aggregates were investigated. The concrete specimens were cured under water, cured under sulphate solution and cured in the freeze and thaw chamber for 1 year. Table 4 summarises the porosity and pore volume of concrete specimens incorporating silica fume and marble dust at 1 year. It should also be noted that the porosity of control specimen cured under water for 28 days was 40.1%. The results shown in Table 4 demonstrated that the incorporation of silica fume along with the increase in the replacement level of marble dust resulted in a significant decrease in both the porosity and pore volume of concrete specimens cured under water for 1 year. It must be noted that the porosity values provided in Table 4 are in a good agreement with Marvila et al. (2019a). When the concrete specimens were cured under sulphate solution and cured in the freeze and thaw chamber, irrespective of the type of replacement level, a substantial increase was observed both in the porosity and pore volume of the concrete specimens. However, it must be noted that even under severe exposure of sulphate attack and under freeze and thaw cycles, concrete specimens incorporating silica fume along with marble dust resulted in a considerable decrease in both the porosity and pore volume of concrete at 1 year. It can therefore be summarised that the use of silica fume in combination with marble dust played a significant role in decreasing the porosity and pore volume of concrete. Decreases in the porosity of the concrete specimens incorporating silica fume and marble dust, particularly under the conditions of sulphate solution and freeze and thaw cycles, validates the enhanced durability associated with these additions. It is also important to note that when compared to concrete containing 20% silica fume alone, the porosity of concrete containing 20% silica fume and 20% marble dust decreased more than ~16% under all the curing conditions examined in the paper. This highlighted the substantial role of marble dust on the improved physical properties of concrete and made significant contributions on the development of mechanical properties and durability characteristics of concrete specimens over the long-term.



#### 4.7 Cost Efficiency Factor

Cost efficiency analysis of concrete incorporating silica fume and marble dust was undertaken using Eq. (1) at 28 days, 1 year and 3 years based on water cured specimens. Calculated cost efficiency factors of concrete incorporating silica fume and marble dust are summarised in Table 5. Results show that incorporating silica fume resulted in a considerable increase in the cost efficiency factor of concrete even at an early age of 28 days. Up to 1 year, there is a slight decrease in the cost efficiency factor of concrete incorporating silica fume and marble dust compared to concrete with silica fume alone. The slight decrease observed in the cost efficiency factor of concrete is mainly responsible from the decrease in compressive strength of concrete incorporating both silica fume and marble dust. Incorporating marble dust in pozzolanic concrete led to a substantial increase in the calcium oxide (CaO) content (shown in Table 3) as well as an overall decrease in the  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$  content in the matrix which is mainly responsible from the decelerating of the pozzolanic reaction (Arel, 2016b). It is shown in Table 5 that cost efficiency factor of concrete incorporating both silica fume and marble dust has increased dramatically in 3 years. The actual performance of pozzolanic concrete incorporating marble dust can only be seen over the long-term (3 years) due to the slow nature of the pozzolanic reaction as well as its dependence on the formation of  $\text{Ca}(\text{OH})_2$ . The cost efficiency factor of concrete increased from 62% to 107% at 3 years as a result of the incorporation of silica fume and marble dust. A substantial increase in the overall performance of concrete played a key role in increasing the cost efficiency factor.

#### 4.8 Cement production and $\text{CO}_2$ emissions

In 2018, Turkey alone produced approximately 72.5 million tons of concrete (Arel, 2016a). In this regard, 1 ton of cement releases 0.94 tons of  $\text{CO}_2$  into the atmosphere (Arel, 2016a). Approximately 68.15 million tons of  $\text{CO}_2$  was released into the atmosphere in 2018 from the cement manufacturing practices in Turkey. The global cement production in 2018 was approximately 5.4 million tons equating to 5.1 billion tons of  $\text{CO}_2$  emissions into the atmosphere. It is also estimated in a previous study (Kim et al., 2013) that producing fine aggregates for concrete results in releases of  $0.08 \text{ kg-CO}_2/\text{m}^3$ .  $\text{CO}_2$  emissions of silica fume,  $\sim 0.014 \text{ ton-CO}_2/\text{t}$  according to the EPA, is also taken into account in this study. Compressive strength of concrete

incorporating silica fume and marble dust examined in this paper are plotted versus the CO<sub>2</sub> emissions in Figure 11. It can be seen in Figure 11 that incorporation of 20% silica fume did not only result in a dramatic decrease in the CO<sub>2</sub> emissions but also enabled a significant increase in the compressive strength of concrete at 3 years. Increasing the marble dust content as a replacement material for fine aggregate further contributed in decreasing the CO<sub>2</sub> emissions as well as increasing the compressive strength of concrete. It must also be noted that the degree in the reduction of CO<sub>2</sub> emissions by utilising marble dust and silica fume in concrete are in a good agreement with cement mortars incorporated gold mine tailings demonstrating the significance of waste utilisation in cement based materials (Ince, 2019). It is also stated in Azevedo et al., (2019) that reusing such wastes in a new industrial product supports a cleaner and environmentally friendly manufacture practices to be developed.

## 5. Conclusions

This paper reports the vital role of waste marble dust incorporation on the long-term mechanical properties and durability characteristics of pozzolanic concrete. The mechanical and physical properties of concrete incorporating waste marble dust, reported in the paper, strongly validates the compatibility of these materials in structural applications in construction practice. It was also demonstrated in the paper that increased sulphate attack and freeze and thaw resistance of concrete incorporating waste marble dust is attained and that the microstructural analysis conducted by Scanning Electron Microscopy were in a very good agreement with these observations. More importantly, the paper reports a substantial improvement in the cost efficiency factor of concrete incorporating waste marble dust validating the degree of potency of such a waste utilisation for the development of sustainable and cost effective construction materials. The utilisation of these industrial wastes in concrete has also been shown to mitigate the CO<sub>2</sub> emissions significantly and hence enabled the improved alternative routes for developing the economic recovery and reducing the environmental pollution to be reconsidered.

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## Figure captions

Figure 1. Particle size distribution of the raw materials

Figure 2. SEM images of (a) cement, (b) marble dust, (c) silica fume

Figure 3. X-ray diffraction patterns for marble dust and silica fume

Figure 4. Compressive strength of silica fume-concrete incorporated with marble dust. ■; 20%SF, ■; 20%SF with 10% MD, □; 20%□F with %20 MD, ●; concrete control at 28 day□

Figure 5. SEM images of specimens cured under water. Cement control (a) 28 days, (b) 1 year, cement with 20%SF (c) 28 days, (d) 1 year, cement with 20%MD (e) 28 days, (f) 1 year, cement with 20%SF and 20%MD (g) 28 days and (h) 1 year

Figure 6. Water penetration depth of concrete incorporated with 20% SF and with 10 and 20% MD. ○; water penetration depth of concrete control □specimen at 28th day□, ●; Ma□□ of ab□orbed water through the application of pressurised water during the experiment, plotted in a secondary axis

Figure 7. Compressive strength loss of concrete subjected to sulphate attack versus immersed time. ■; concrete control, ■; concrete with 20%□F, ■; concrete with 20%□F and 10% MD, □; concrete with 20%SF and 20% MD. Expansion of concrete subjected to sulphate attack is plotted in a secondary axis, ●; concrete control, ○; concrete incorporated with 20%□F and 20%MD

Figure 8. SEM images of specimens exposed to sulphate testing. Cement (a) 28 days, (b) 1 year, Cement with 20%SF (c) 28 days, (d) 1 year, cement with 20%SF and 20%MD (e) 28 days, (f) 1 year

Figure 9. Compressive strength loss of concrete subjected to freeze and thaw versus time. ■, concrete control; ■, concrete with 20%SF; ■, concrete with 20%□F and 10% MD; □, concrete with 20%SF and 20% MD. ●, Mass loss of concrete subjected to freeze/thaw cycling. ●, concrete control; ○, concrete incorporated with 20%SF and 20%MD

Figure 10. SEM images specimens exposed to freeze and thaw cycling. Cement (a) 28 days, (b) 1 year, cement with 20%SF (c) 28 days, (d) 1 year, cement with 20%SF and 20%MD (e) 28 days, (f) 1 year

Figure 11. Compressive strength (■) versus CO<sub>2</sub> emissions (●) of concrete incorporated with 20% SF and with 10 and 20% MD.

### Table captions

Table 1. Physical properties of raw materials

Table 2. Mix constituents

Table 3. Elemental oxide composition of the raw materials determined by X-ray fluorescence spectroscopy

Table 4. Porosity and pore volume of concrete specimens incorporating silica fume and marble dust under various curing conditions

Table 5. Cost efficiency of concrete incorporating silica fume and marble dust



Table 1: Physical properties of raw materials

Property	Cement	Silica fume	Marble dust
Specific gravity	3.15	2.7	2.6
Loss on ignition	0.3	5.2	38.7
Insoluble residue	0.34	0.64	0.91
Blaine specific surface area (cm <sup>2</sup> /g)	3540	17540	18203

Table 2: Mix constituents

Mixture type	Mixture name	Silica Fume (% by mass)	Marble Dust (% by mass)	w/c ratio	Constituents (kg)					
					Water	Cement	Silica Fume	Marble Dust	Fine Aggregate	Coarse Aggregate
	Control	0	0	0.42	193	460	0	0	625	1082
Cement replacement	20SF	20	0	0.42	193	368	92	0	625	1082
Cement and sand replacement	20SF+10MD	20	10	0.42	193	368	92	62.5	562.5	1082
Cement and sand replacement	20SF+20MD	20	20	0.42	193	368	92	125	500	1082

Table 3: Elemental oxide composition of the raw materials determined by X-ray fluorescence spectroscopy

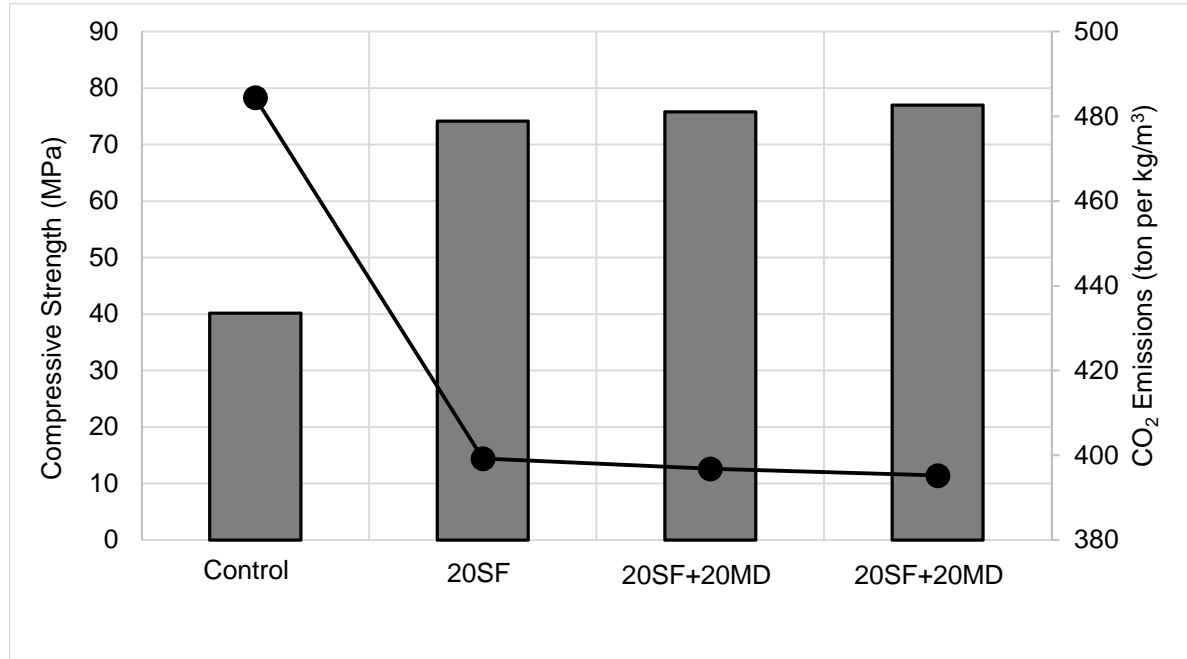
Chemical Composition	Cement (wt%)	Silica Fume (wt%)	Quartz sand (wt%)	Marble Dust (wt%)
CaO	43.8	1.25	0.46	64.5
ZnO	0.01	0.02	-	0.01
SiO <sub>2</sub>	30.2	93.3	91.9	23.4
Al <sub>2</sub> O <sub>3</sub>	11.4	0.73	3.91	4.32
Fe <sub>2</sub> O <sub>3</sub>	5.11	1.82	0.65	2.83
SO <sub>3</sub>	3.29	0.22	0.04	0.47
MgO	2.61	0.89	0.14	1.73
K <sub>2</sub> O	1.34	1.21	1.31	1.04
TiO <sub>2</sub>	0.74	-	0.83	0.48
Na <sub>2</sub> O	0.51	0.28	0.49	0.79
MnO	0.43	0.09	0.04	0.07
Cl	0.03	0.04	0.01	0.07

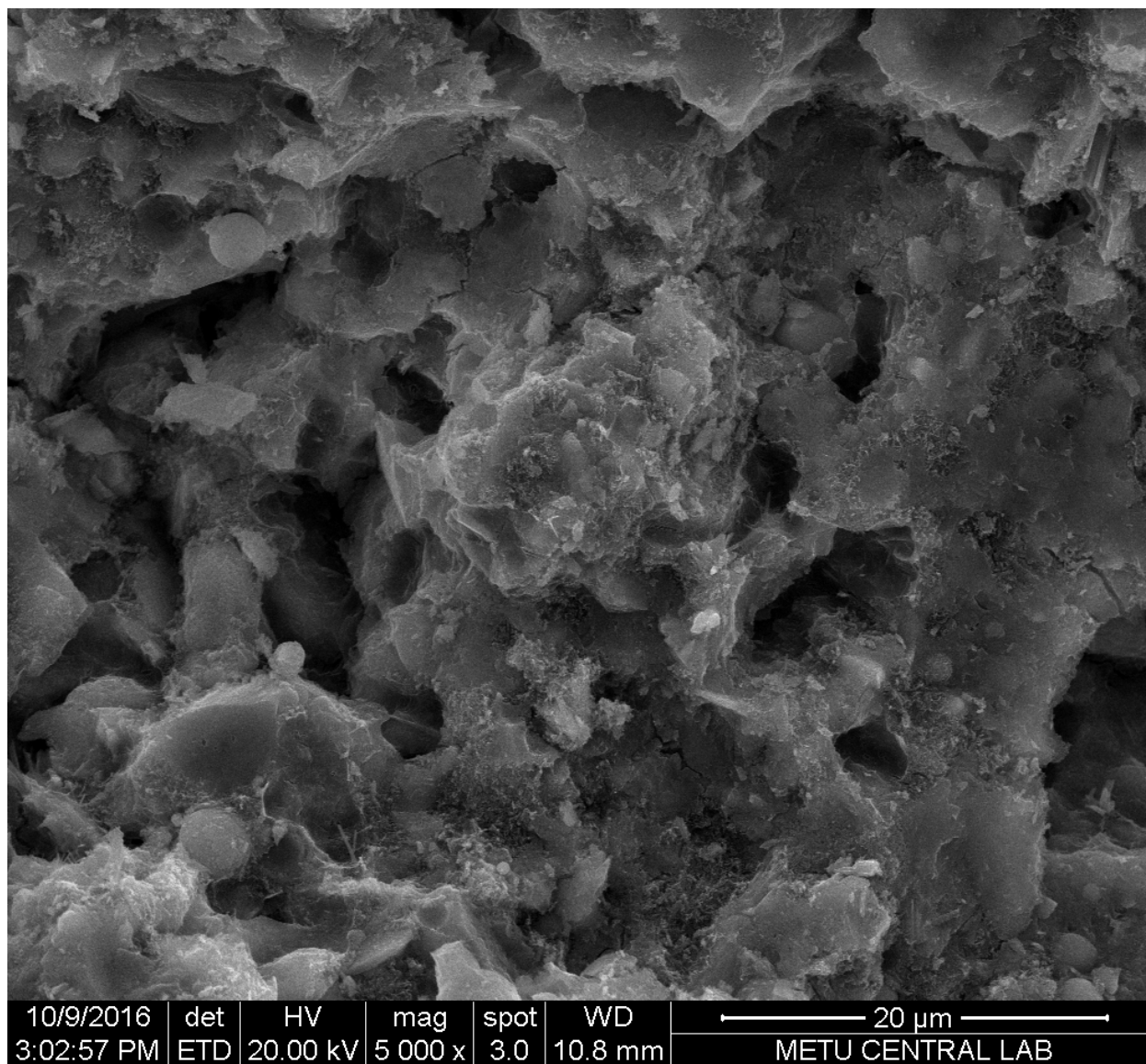
Table 4: Porosity and pore volume of concrete specimens incorporating silica fume and marble dust under various curing conditions

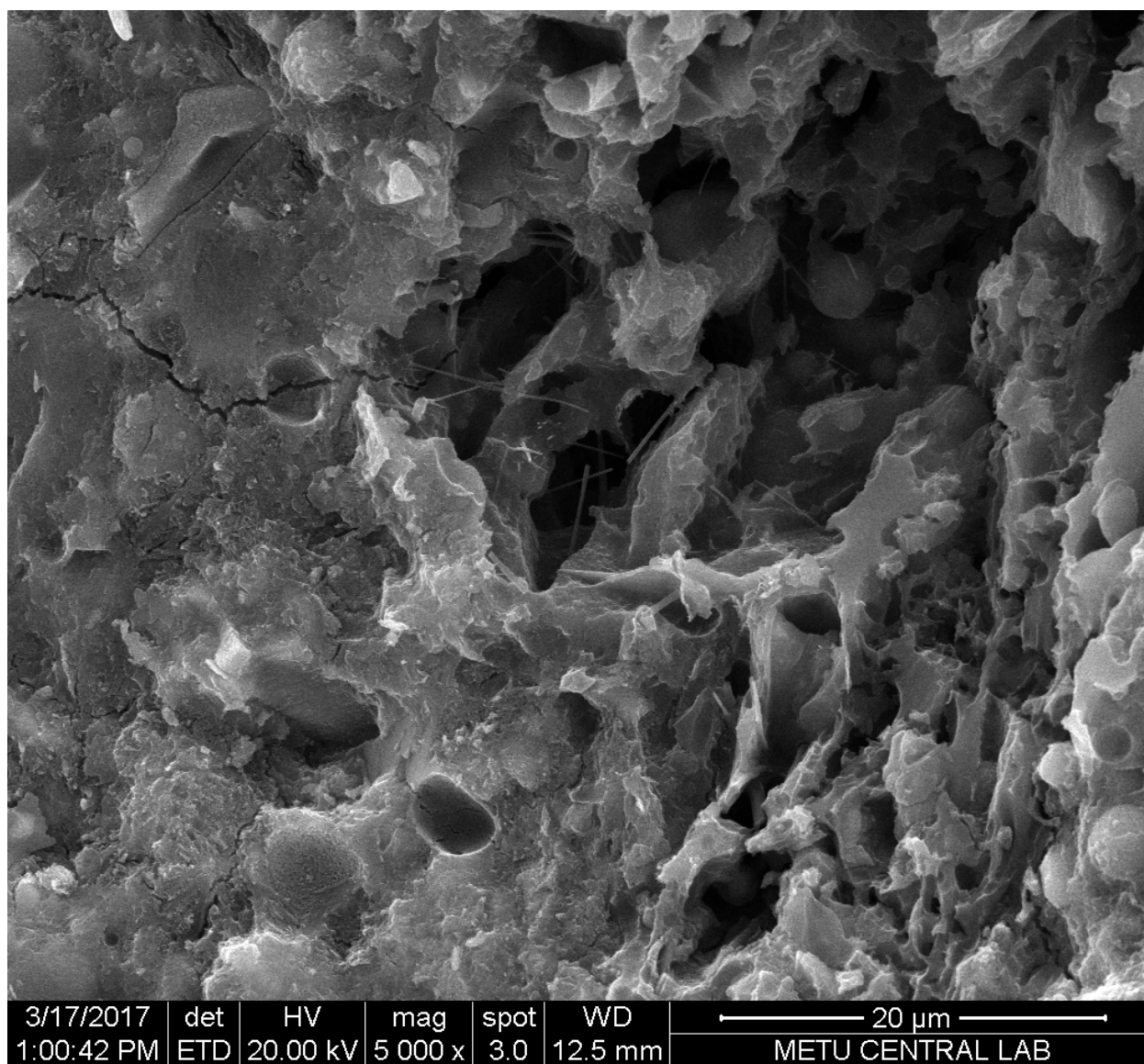
Mixture	Cure Condition	Porosity (%)	Pore Volume (cm <sup>3</sup> )
Cement	Water cured	39.2	0.2814
Cement + %20 SF	Water cured	30.8	0.2207
Cement + %20 SF + %10 MD	Water cured	28.4	0.2123
Cement + %20 SF + %20 MD	Water cured	25.3	0.1659
Cement	Sulphate solution	47.1	0.4365
Cement + %20 SF	Sulphate solution	34.5	0.2571
Cement + %20 SF + %20 MD	Sulphate solution	28.7	0.2145
Cement	Freeze and thaw	49.3	0.4584
Cement + %20 SF	Freeze and thaw	36.6	0.2612
Cement + %20 SF + %20 MD	Freeze and thaw	30.4	0.2194

Table 5: Cost efficiency of concrete incorporating silica fume and marble dust

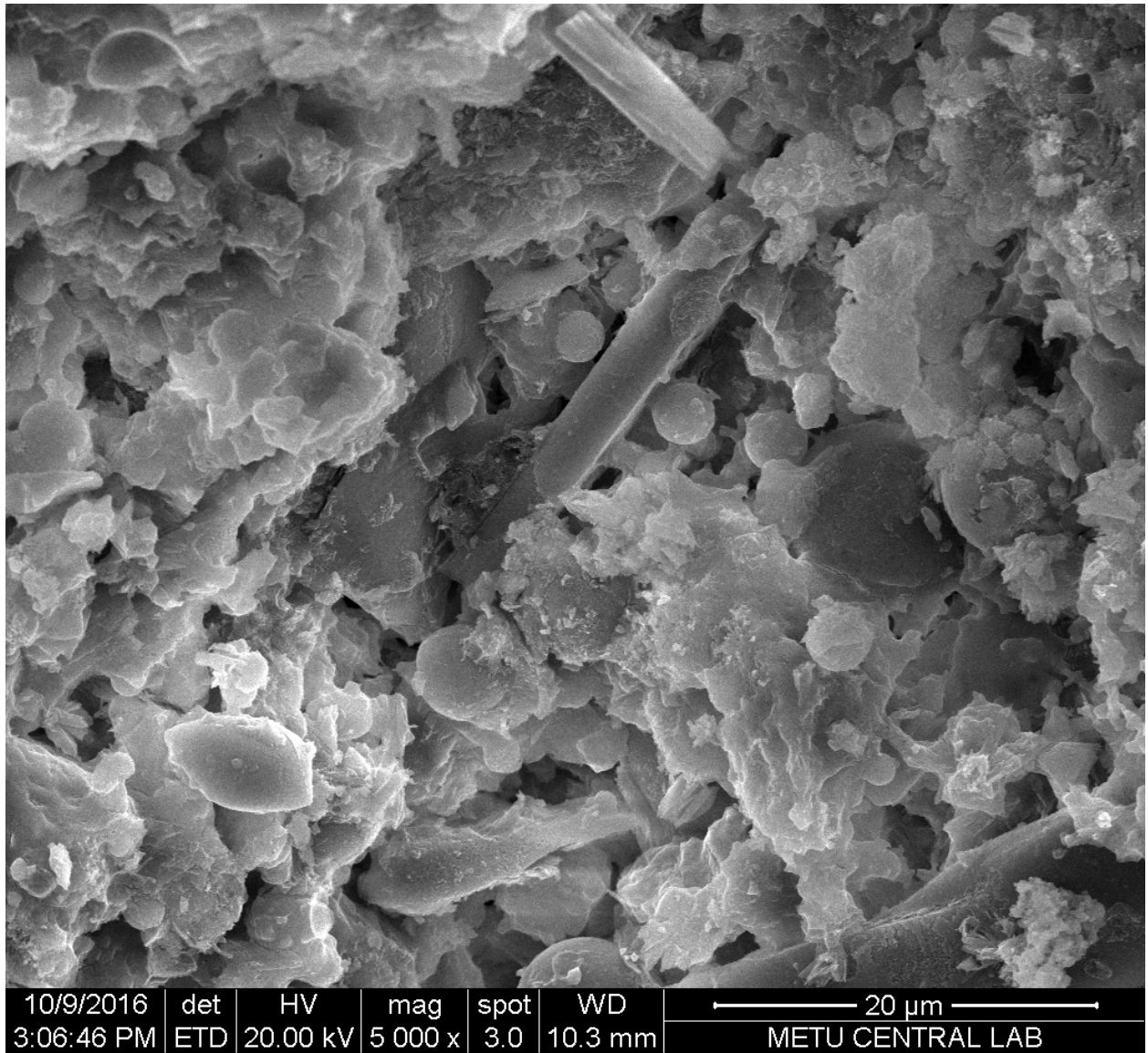
Mix		Mix Constituents				Mix Cost				CEF						
Marble Dust Replacement level (%)	Silica Fume Replacement level (%)	Cement (kg)	Fine aggregate (m <sup>3</sup> )	Coarse aggregate (m <sup>3</sup> )	Silica fume (kg)	Cement (\$)	Fine aggregate (\$)	Coarse aggregate (\$)	Silica fume (\$)	$f_{c_{28d}}$	$F_{c_{1y}}$	$F_{c_{3y}}$	Cost/m <sup>3</sup> (\$)	CEF <sub>28d</sub>	CEF <sub>1y</sub>	CEF <sub>3y</sub>
0	0	460	0.24	0.41	0	55.20	3.56	5.68	0	40.15	40.15	40.15	64.44	62.31	62.31	62.31
0	20	368	0.24	0.41	92	44.16	3.56	5.68	19.32	53.93	66.88	74.15	72.72	74.16	91.97	101.97
10	20	368	0.21	0.41	92	44.16	3.12	5.68	19.32	52.44	62.10	75.78	72.28	72.55	85.92	104.84
20	20	368	0.19	0.41	92	44.16	2.82	5.68	19.32	50.87	58.17	77.02	71.98	70.67	80.81	107.00

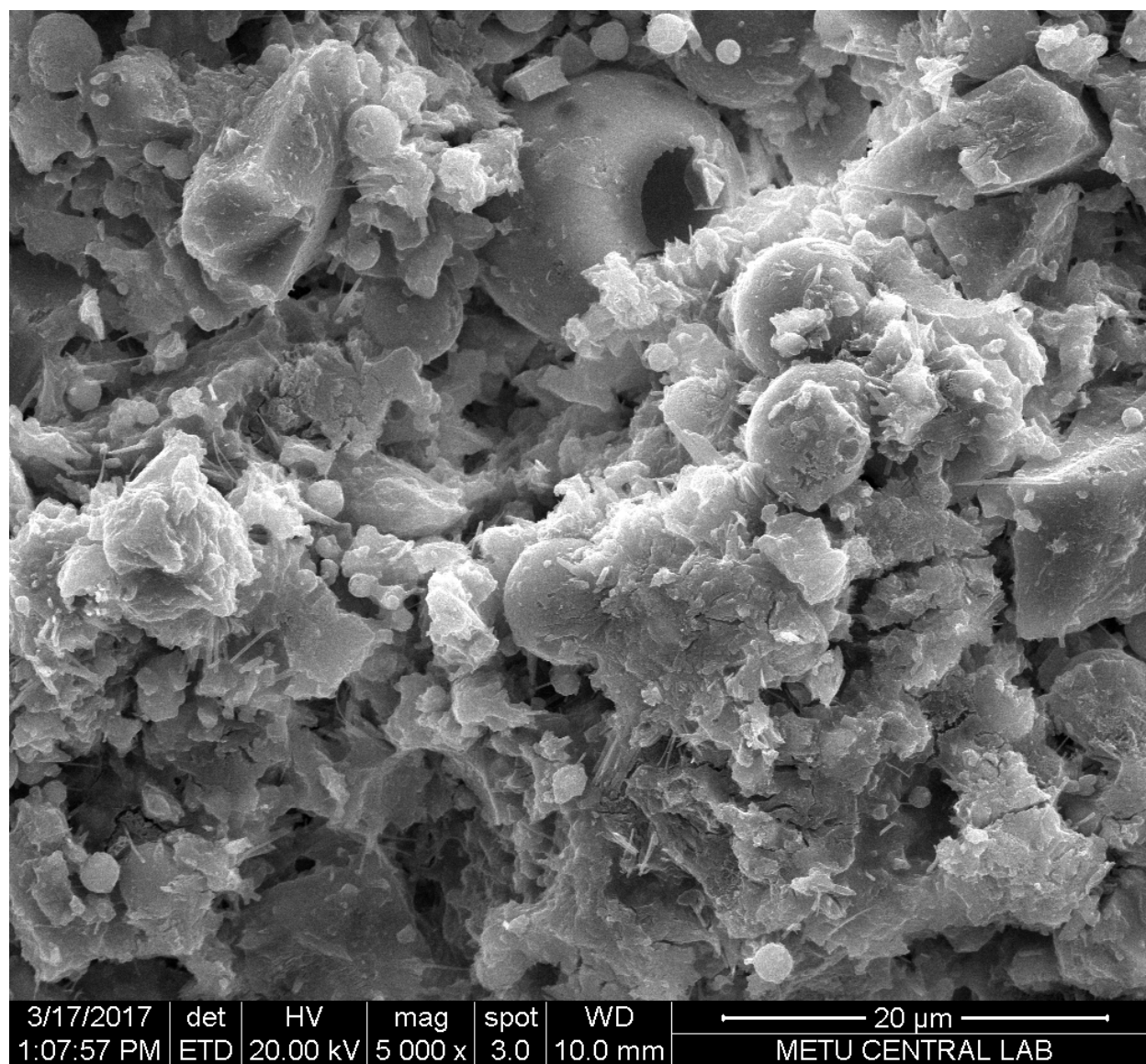




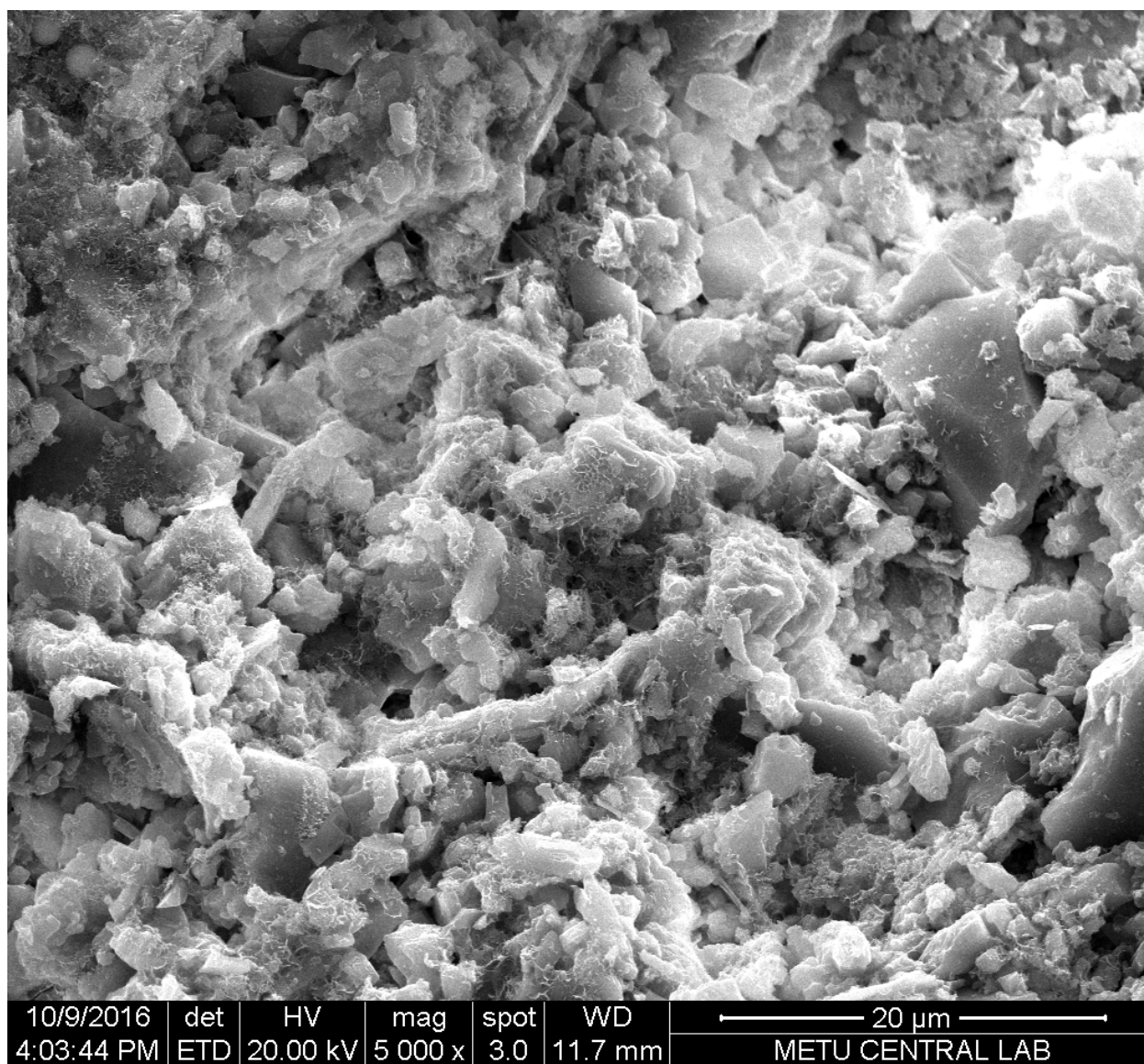


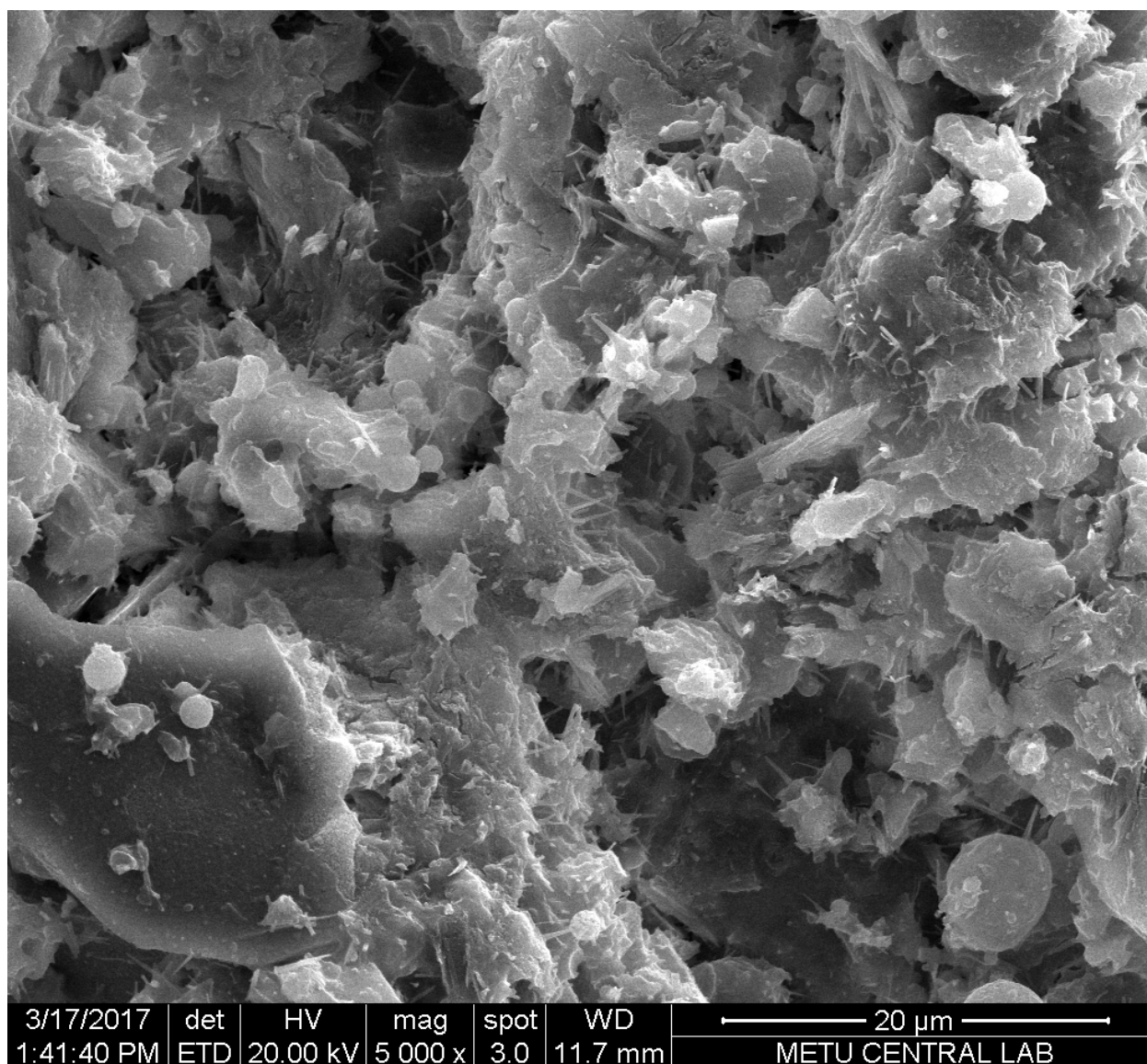


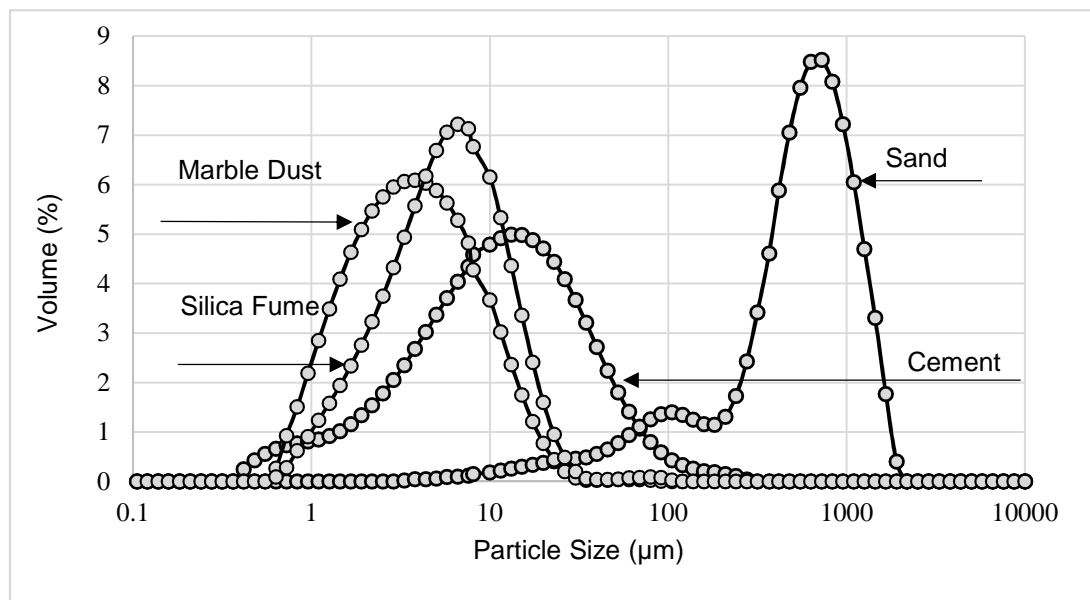




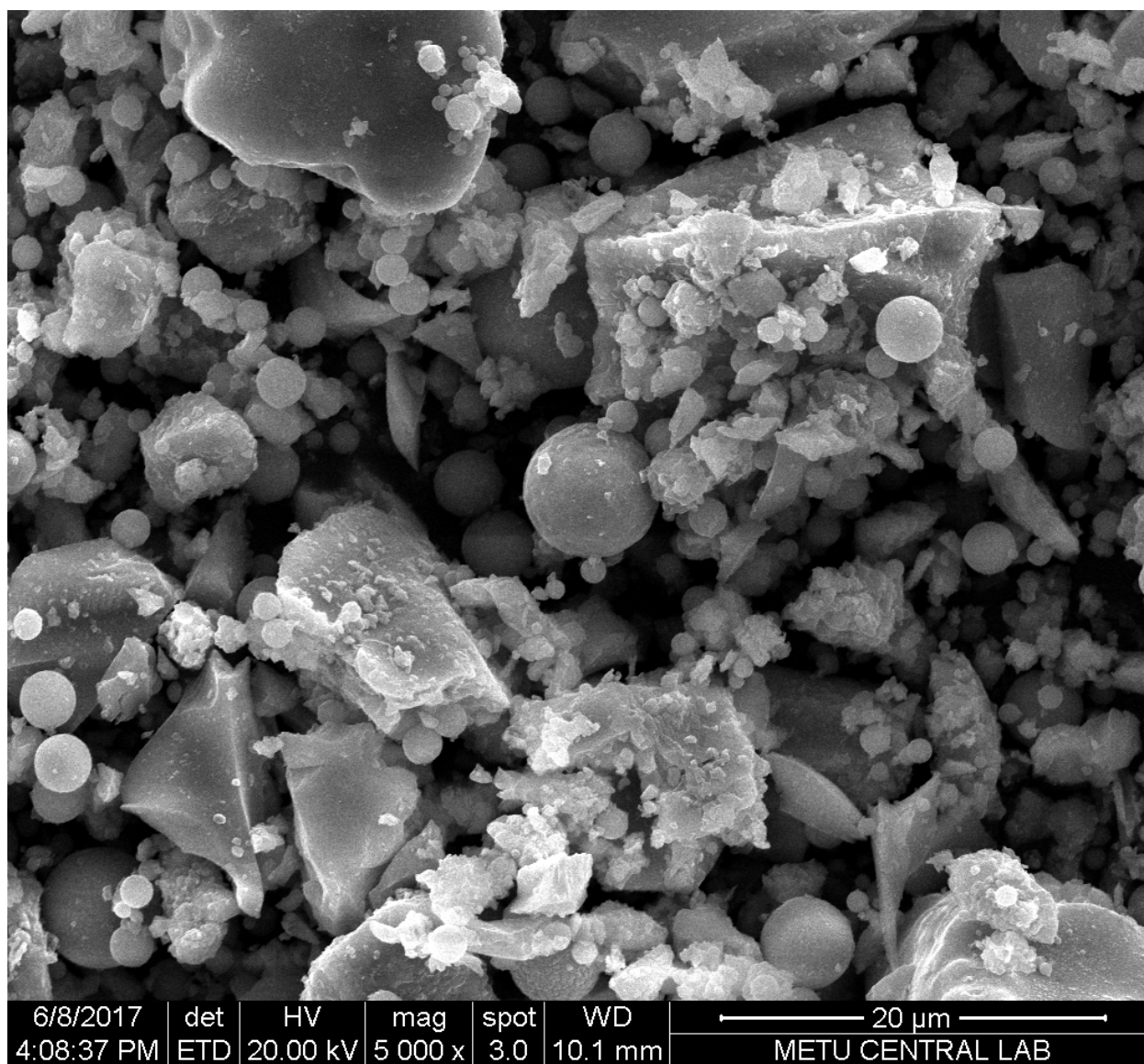


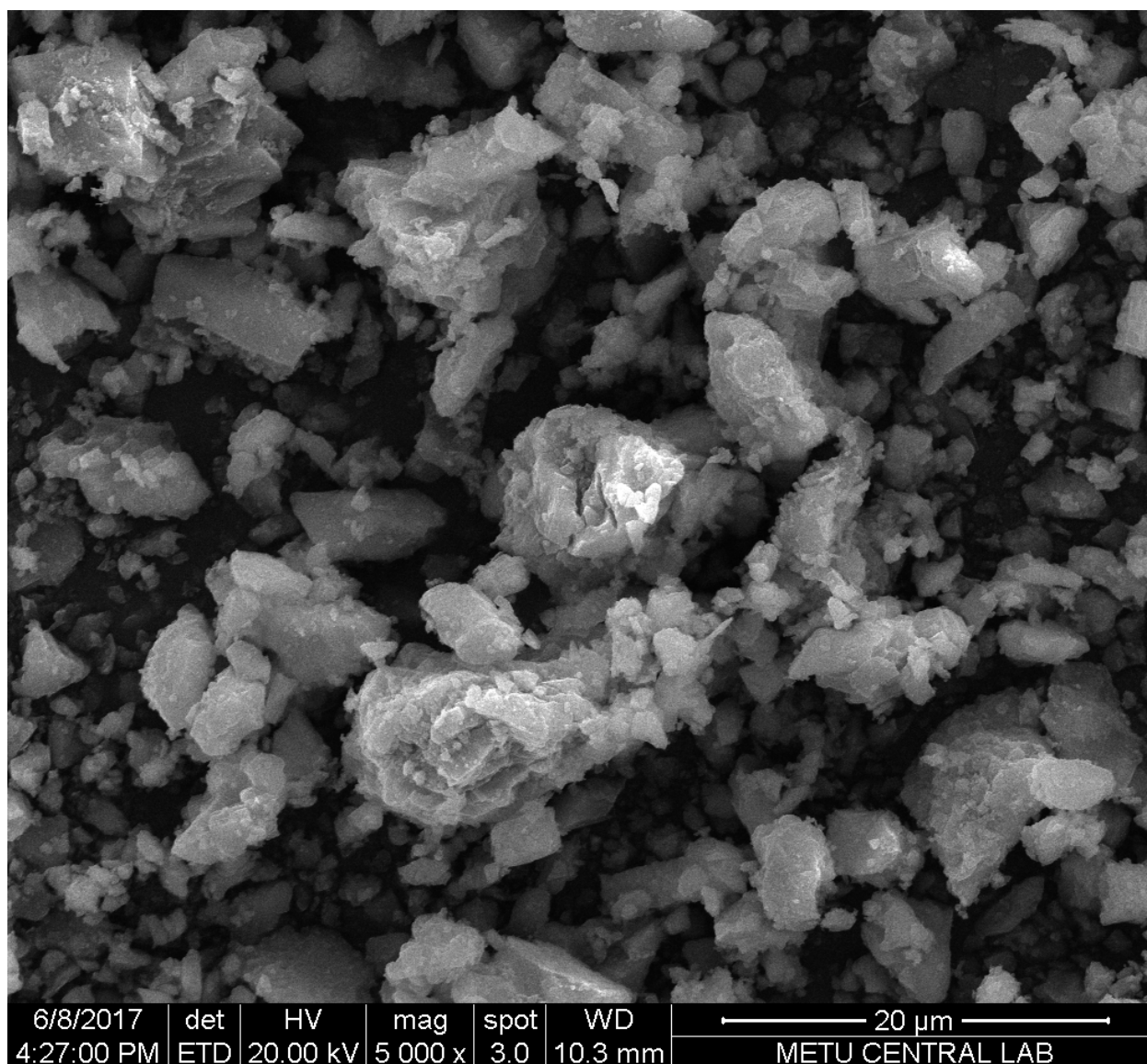




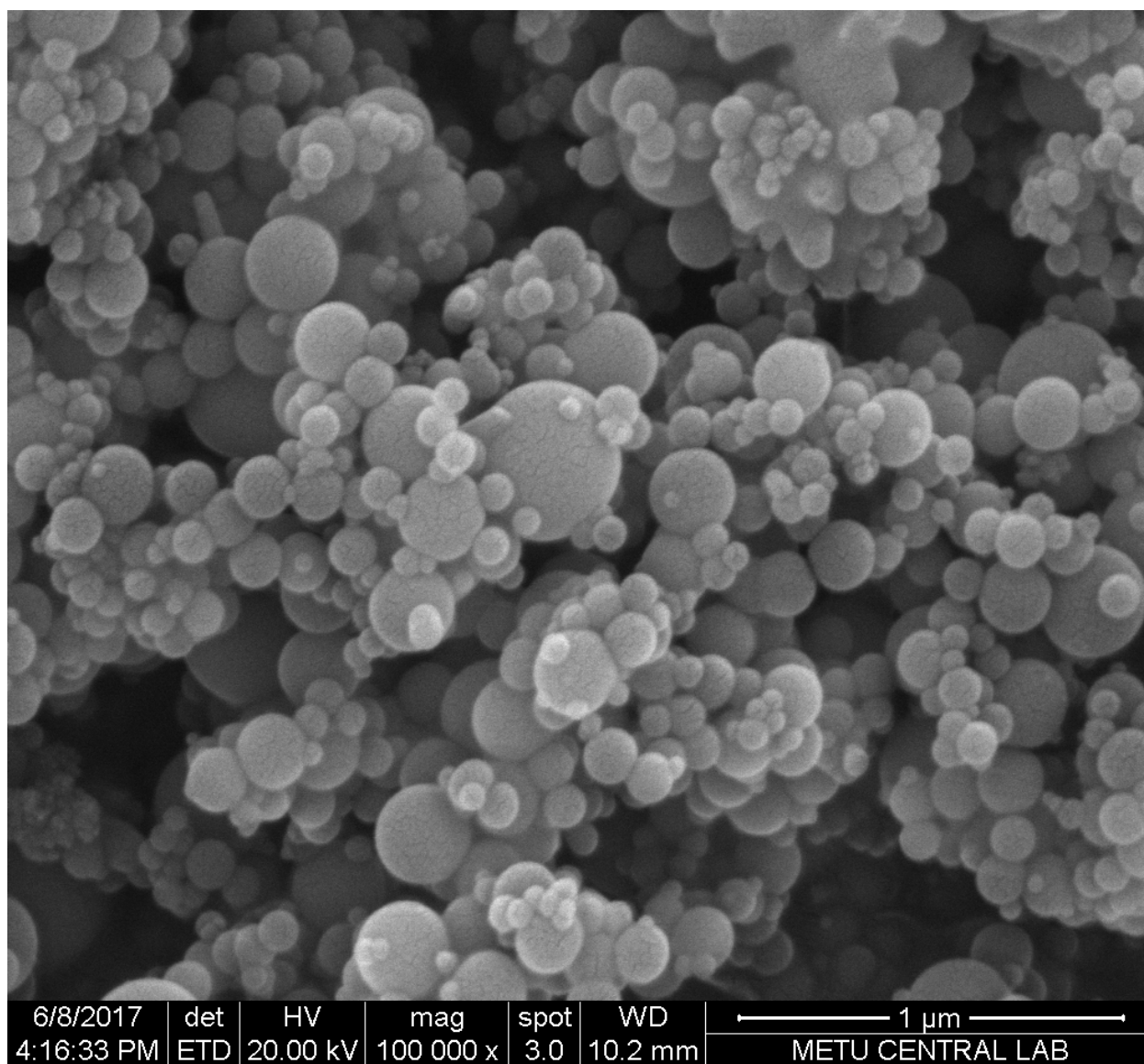




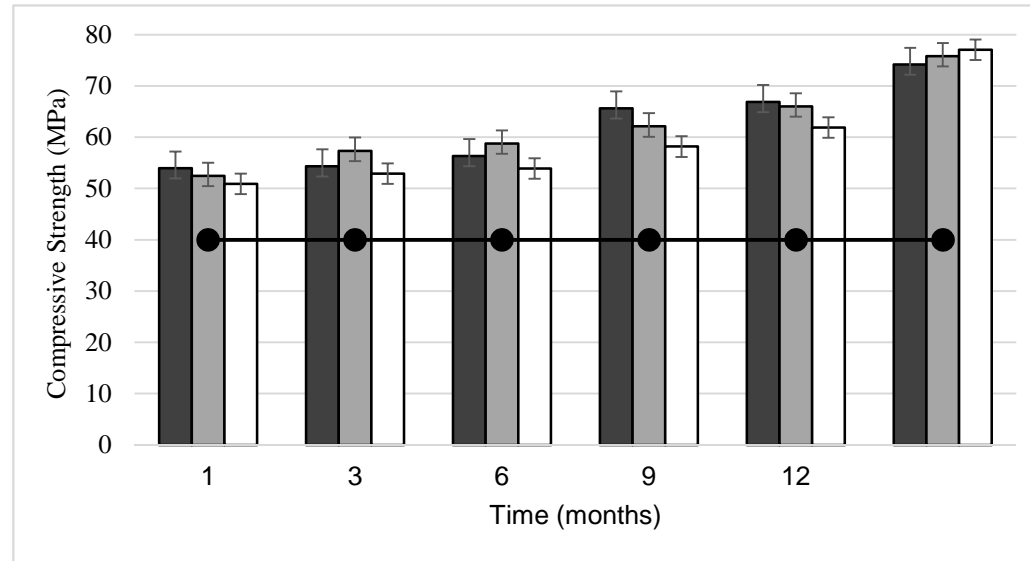


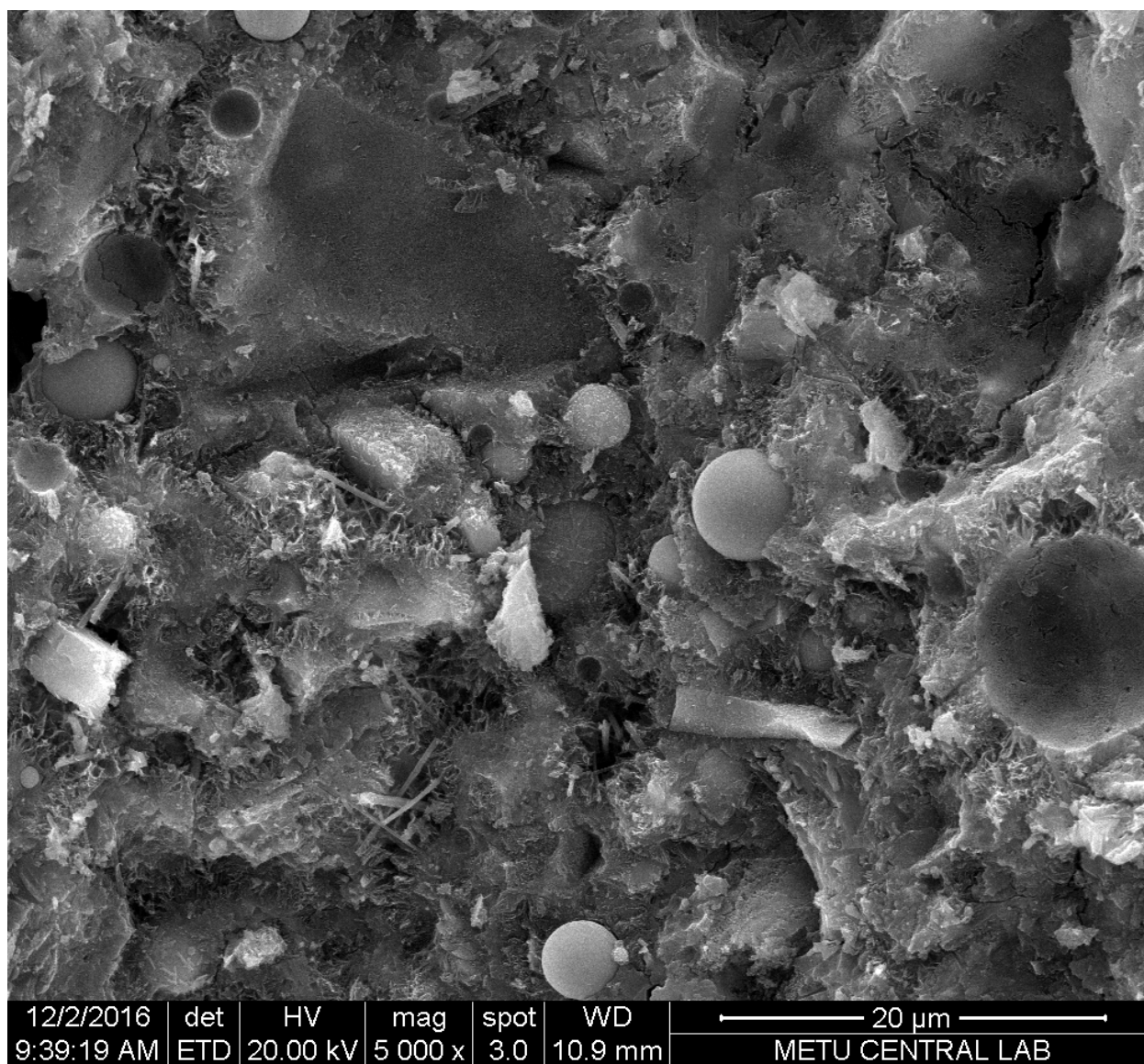


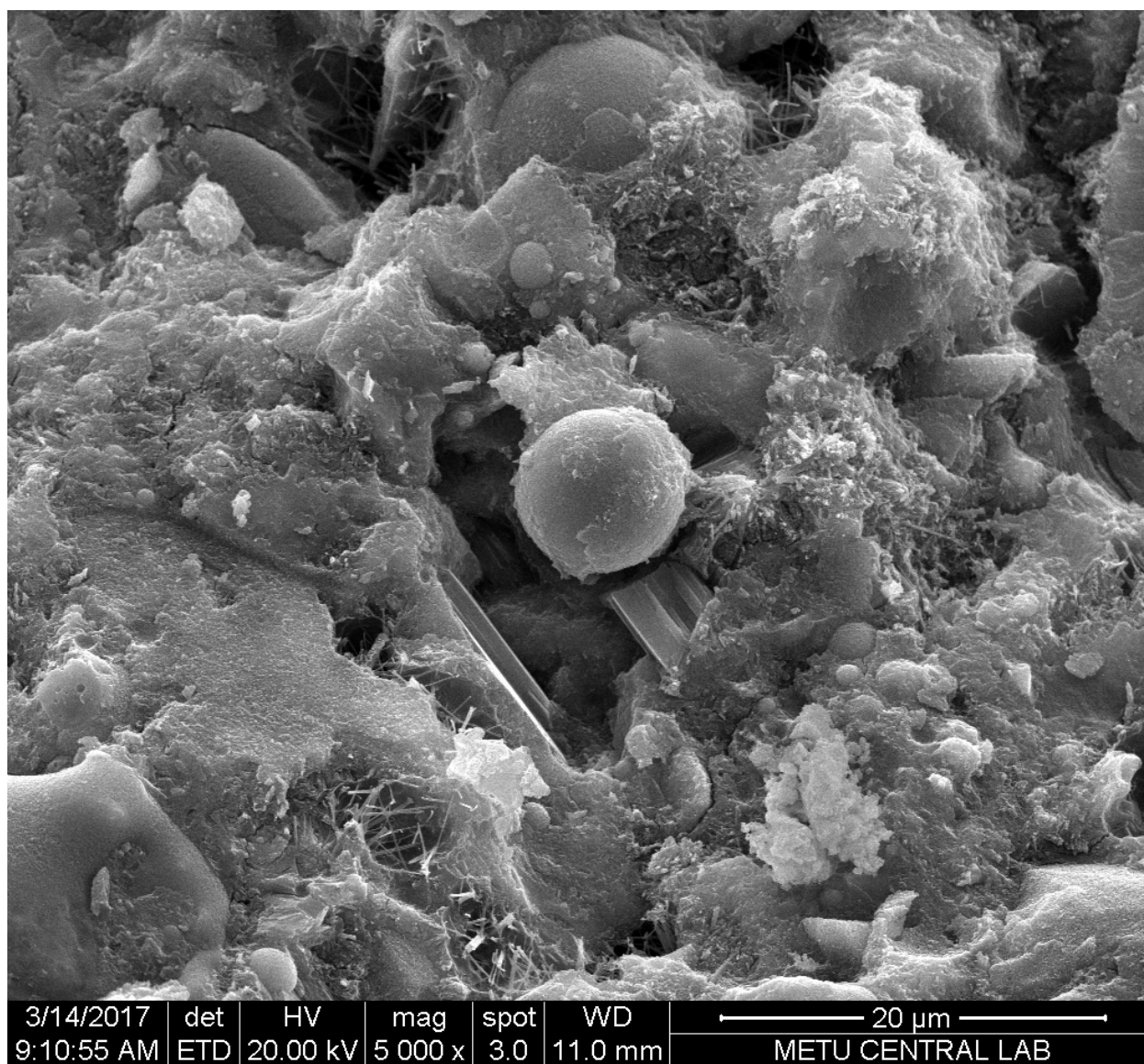




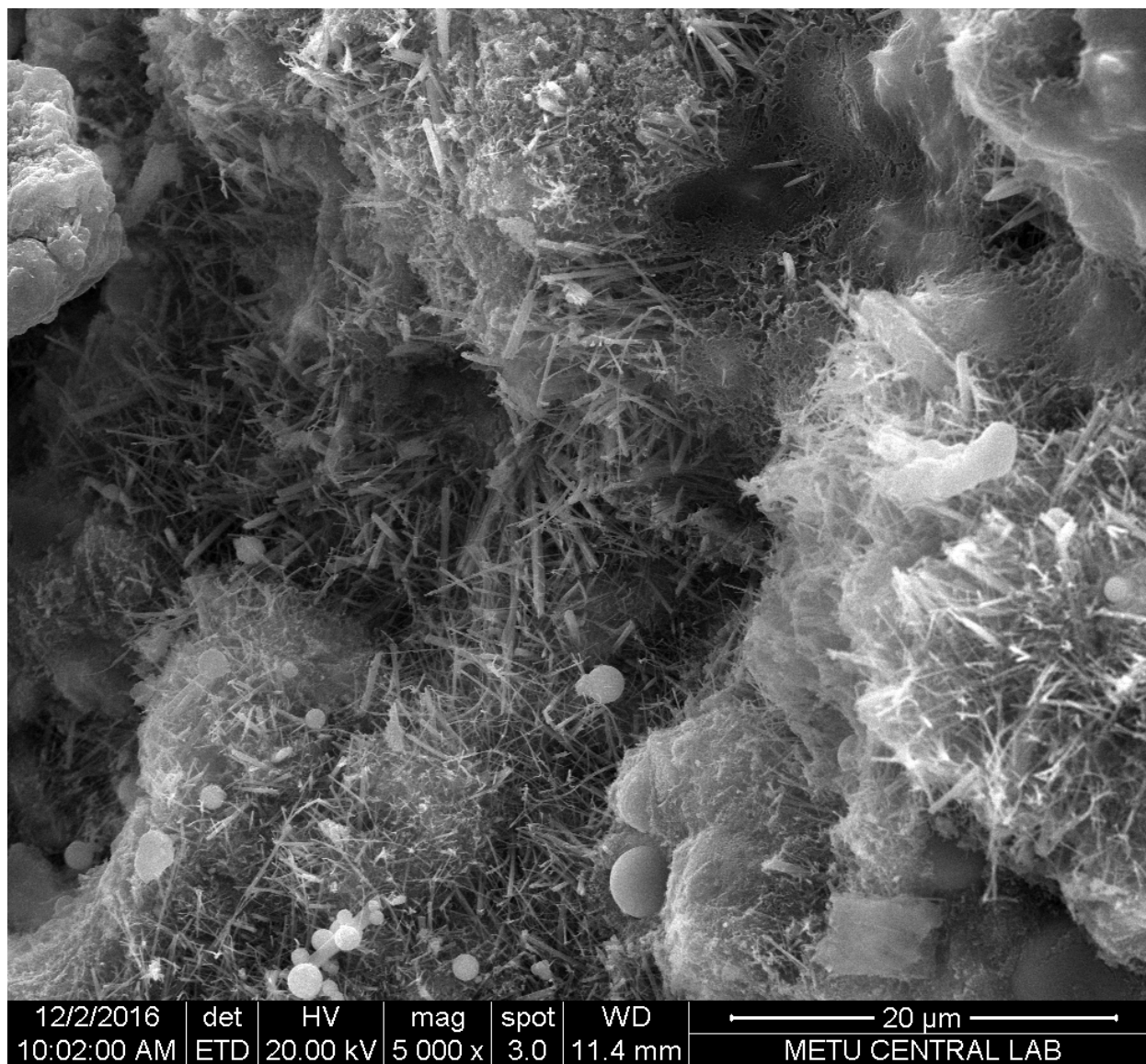


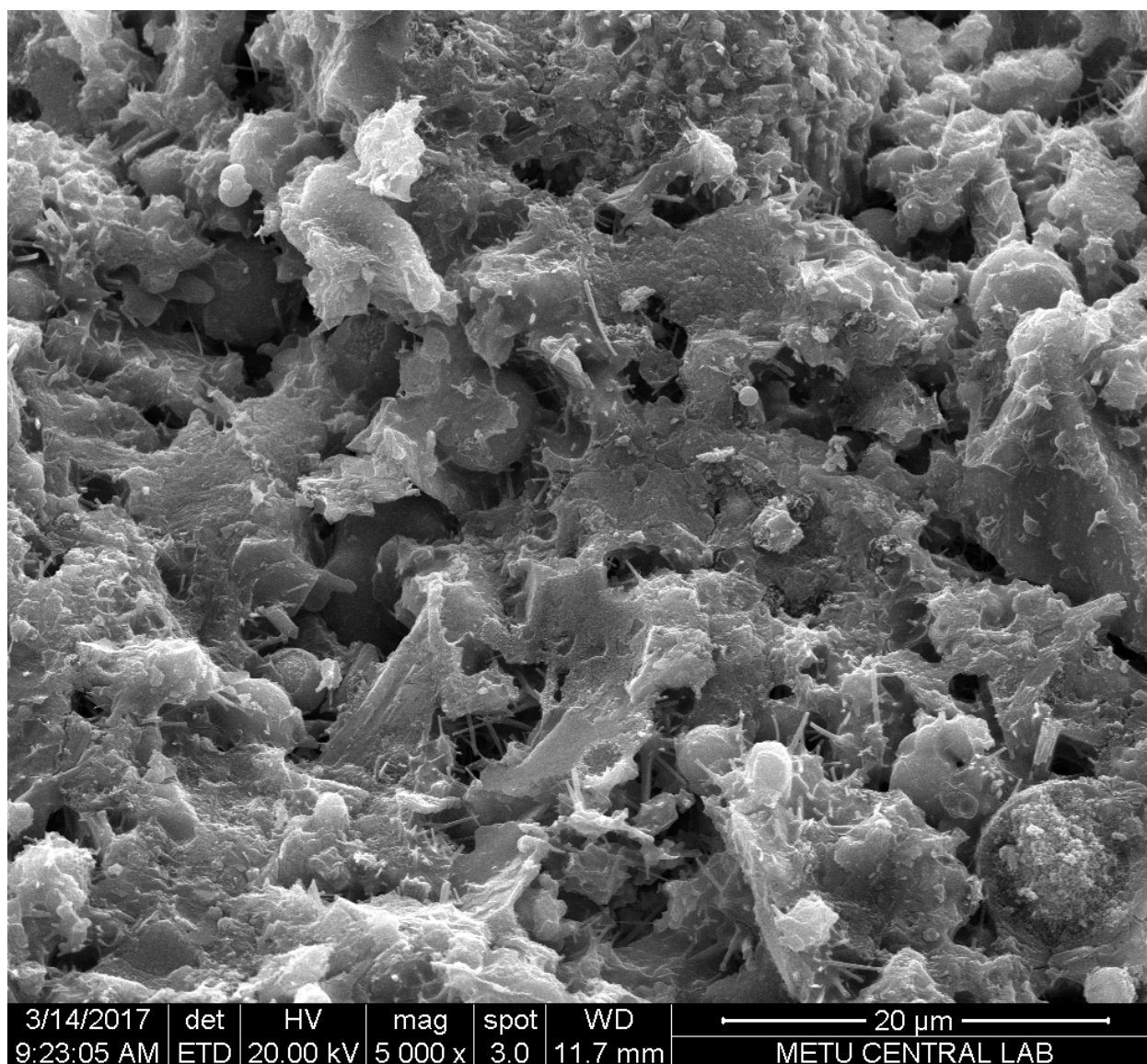




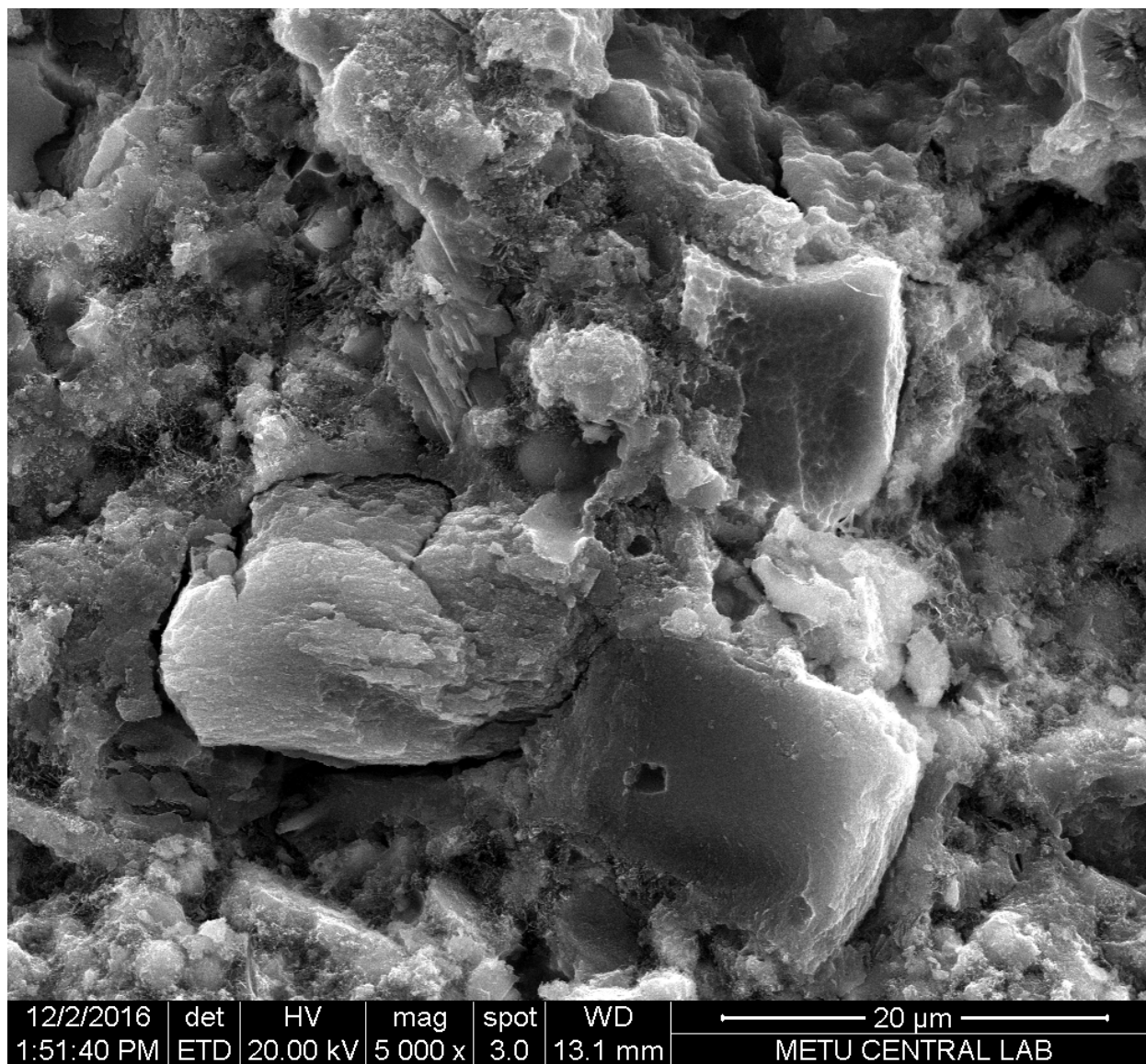


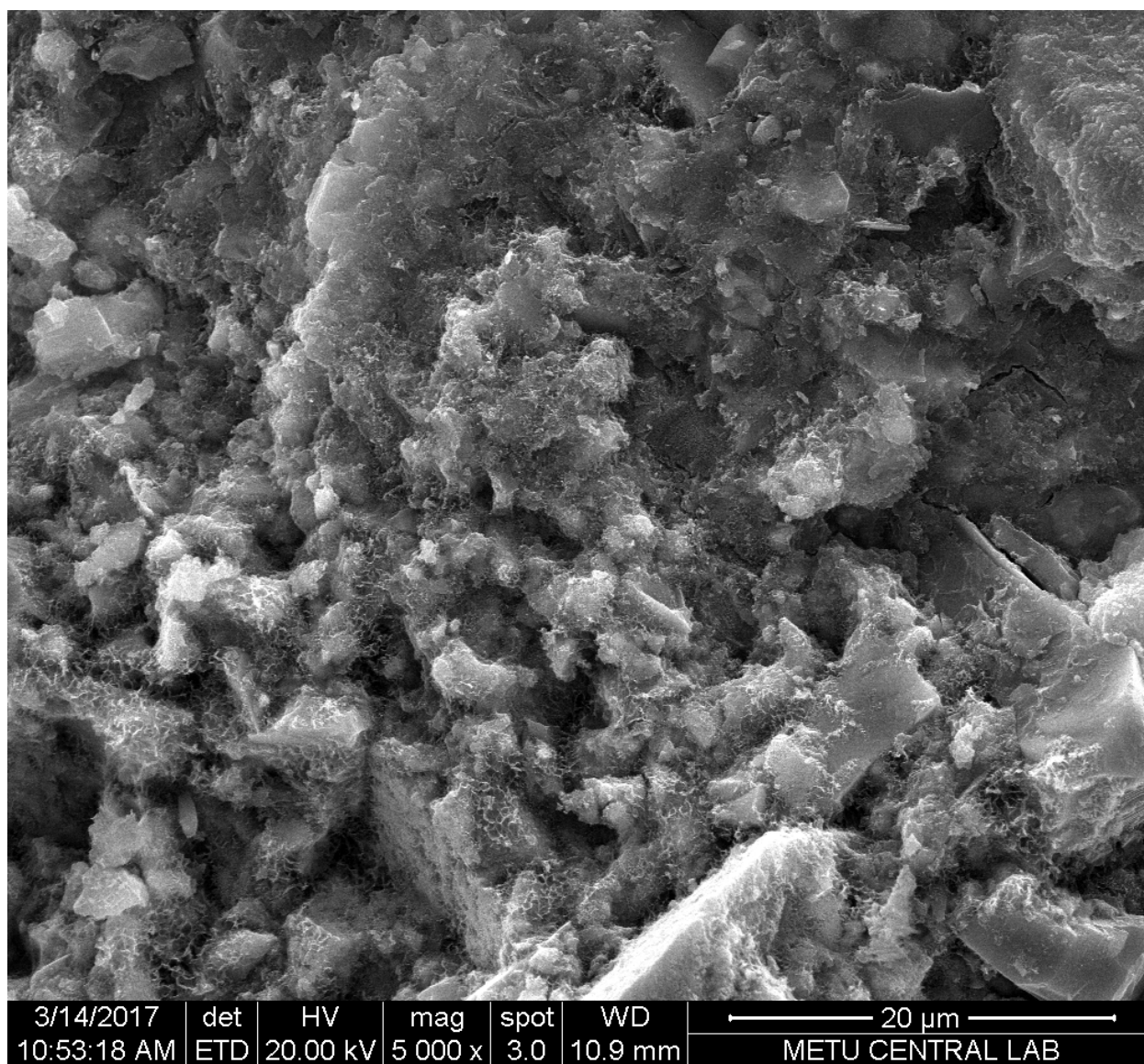




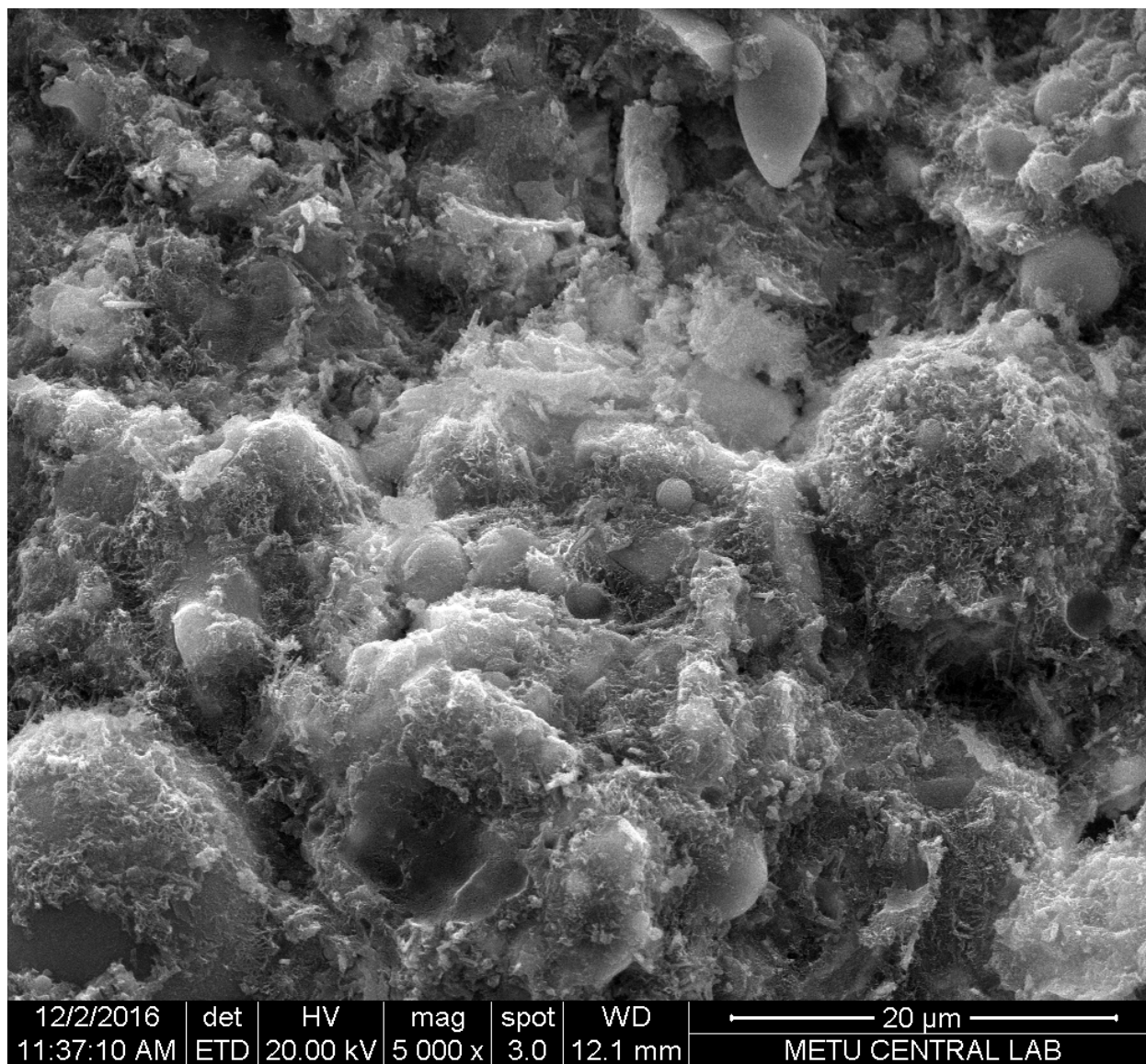




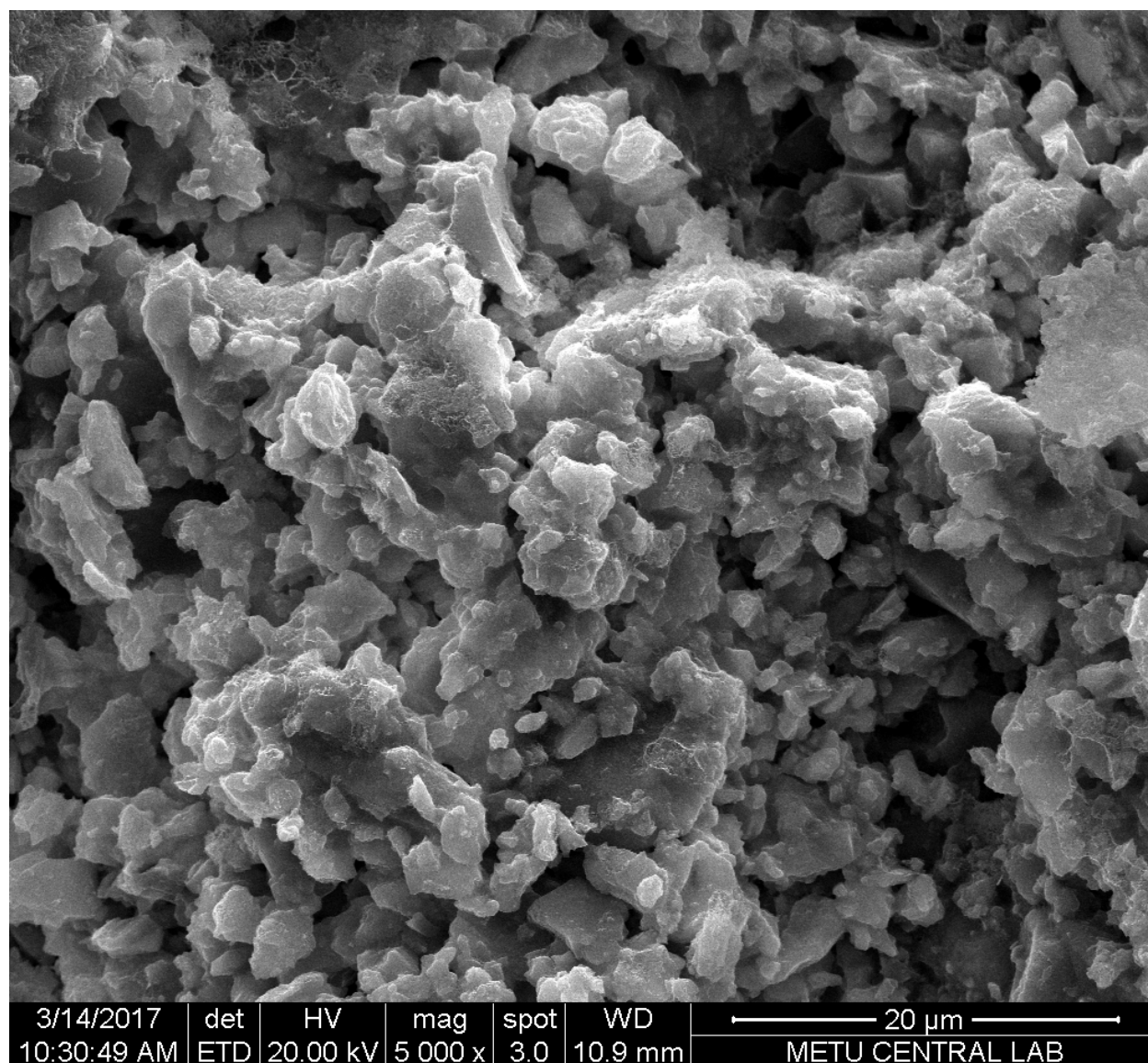


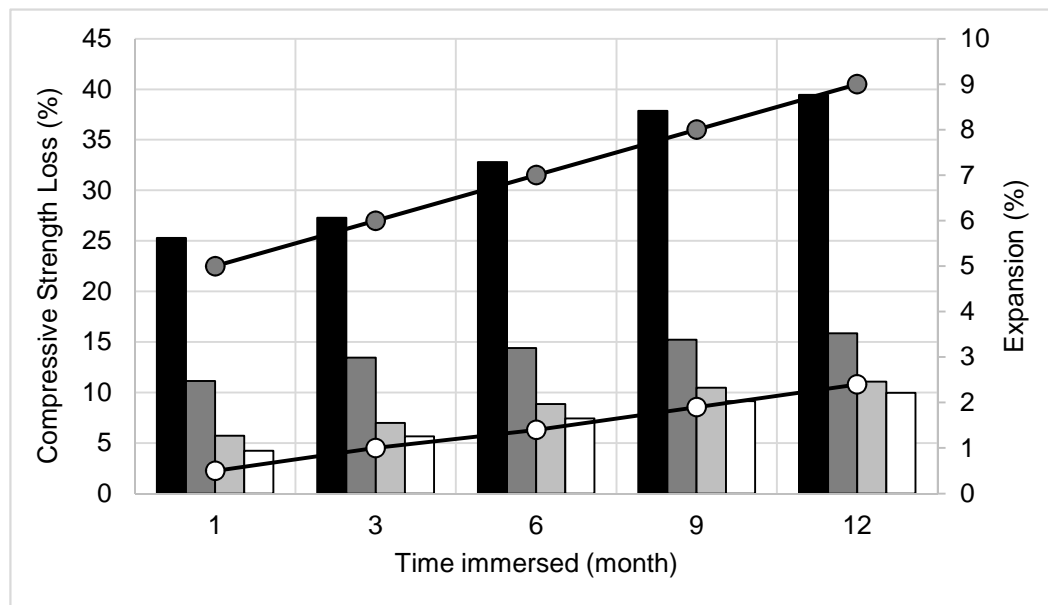


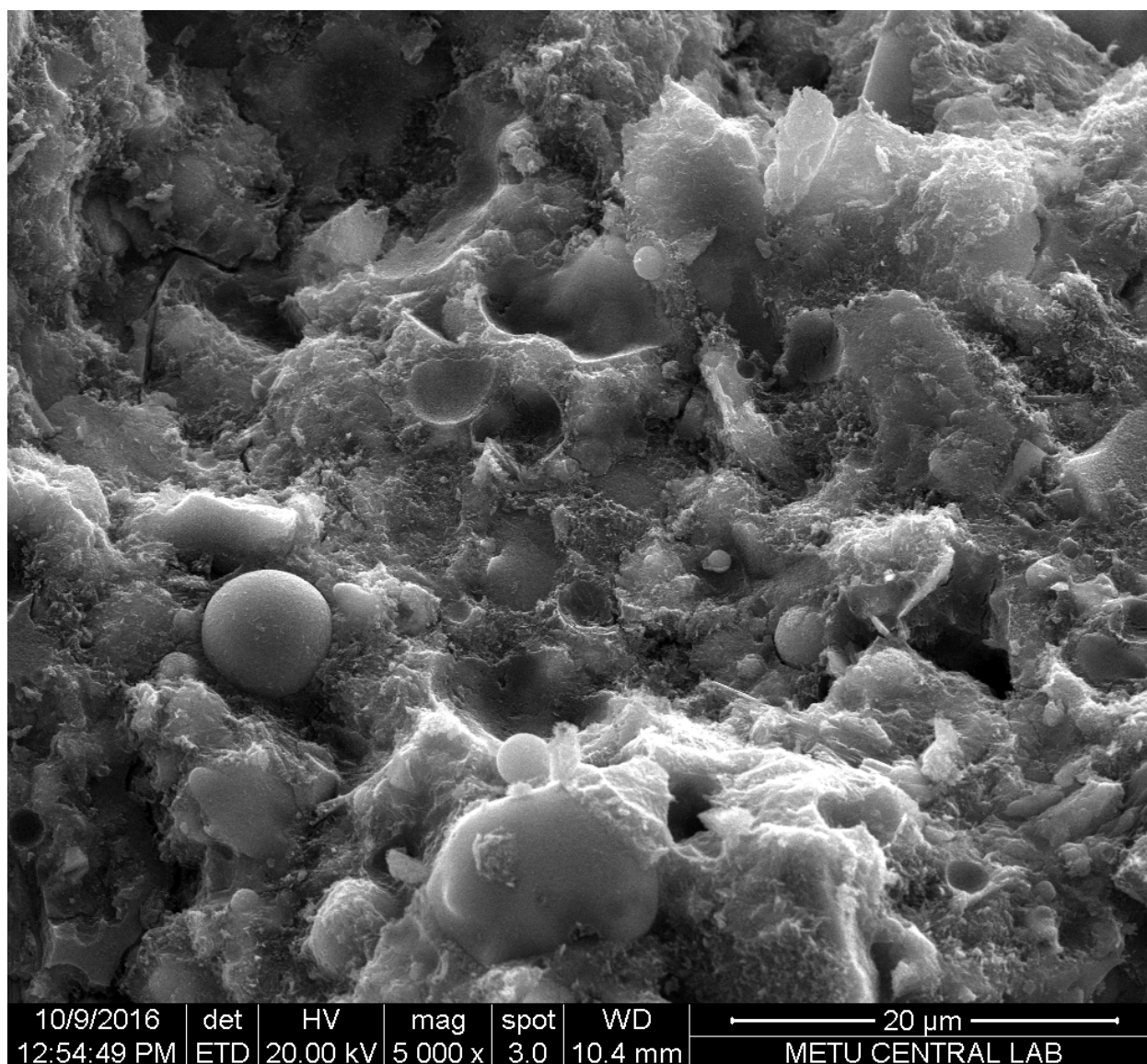




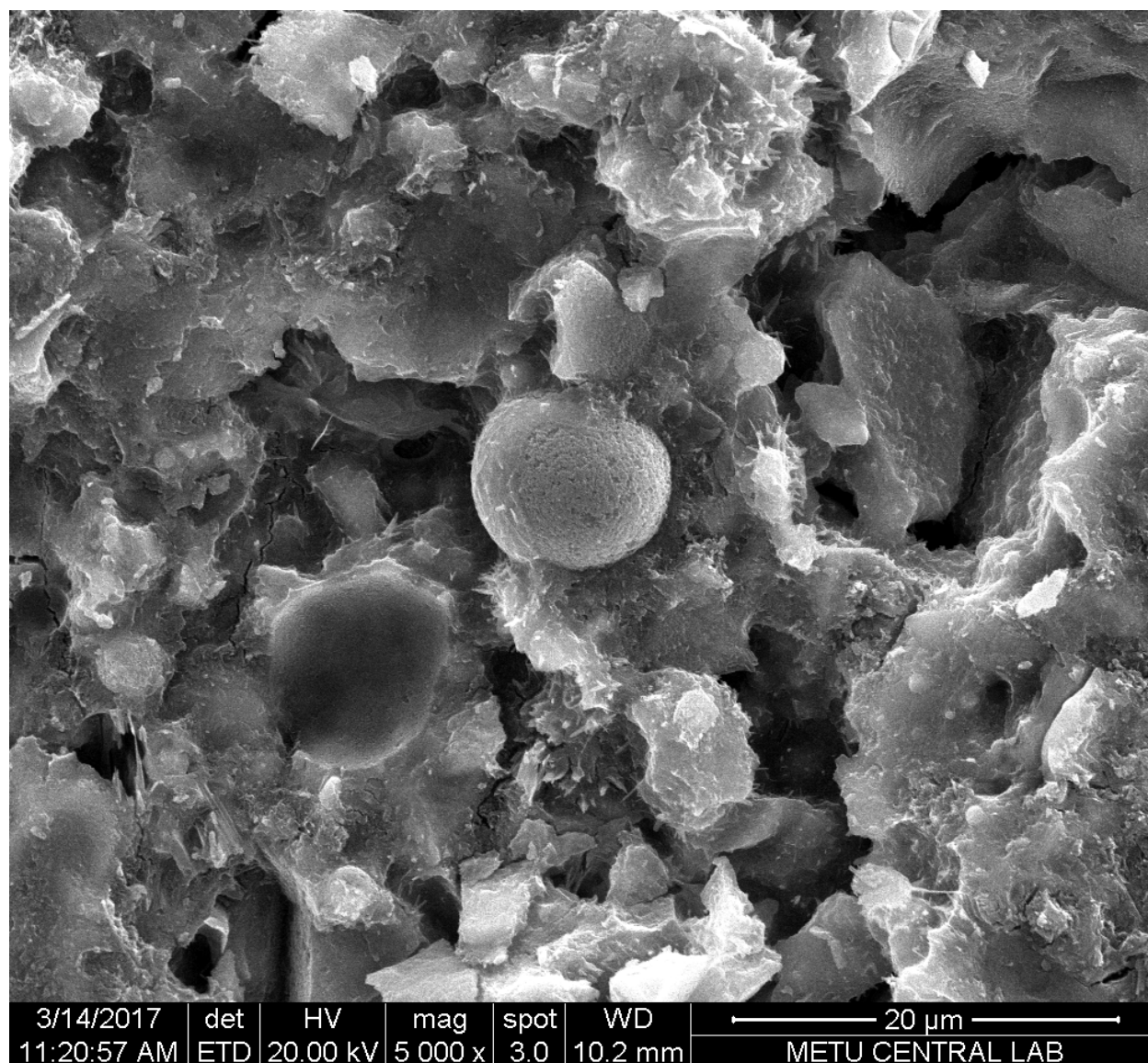


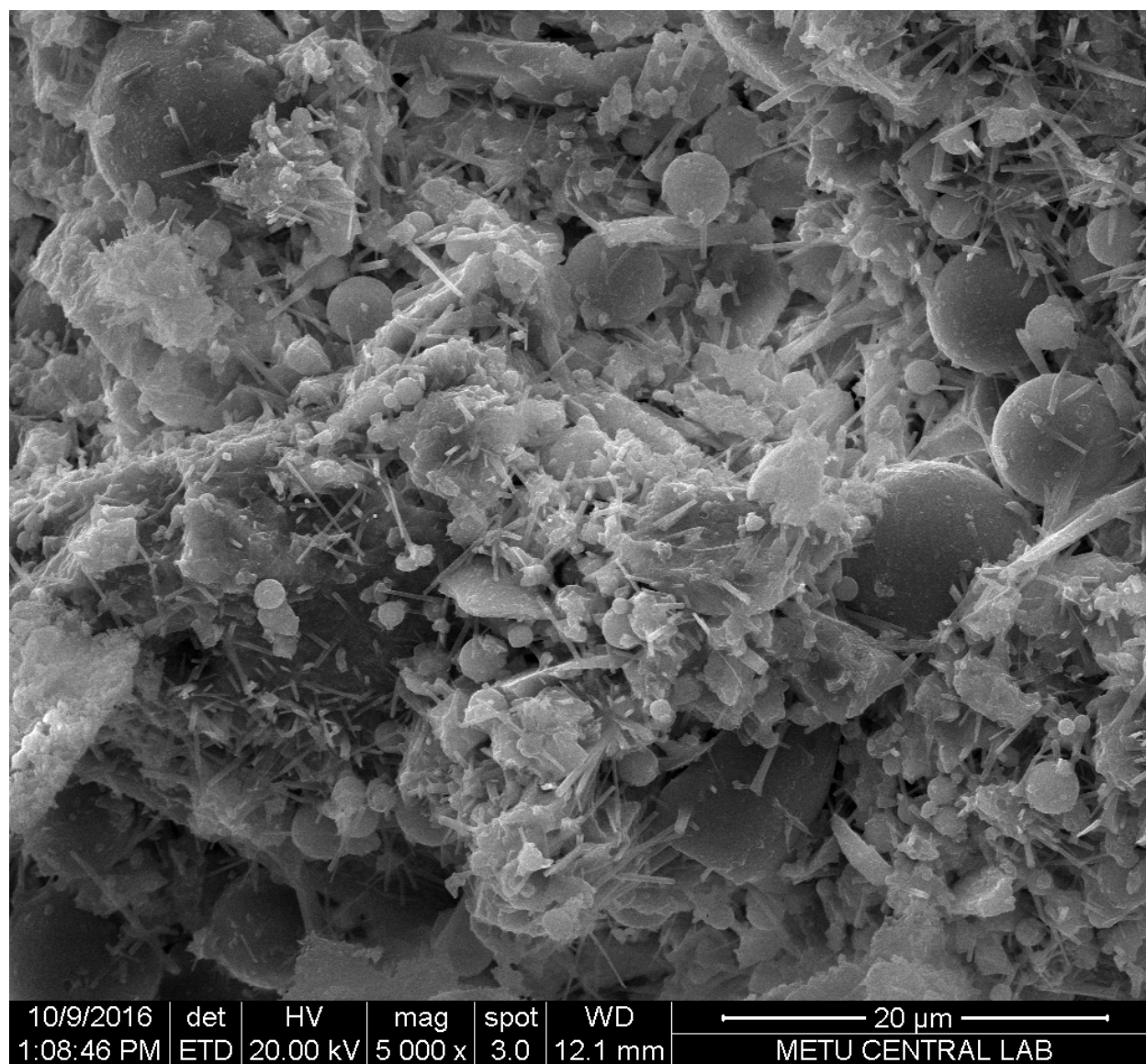




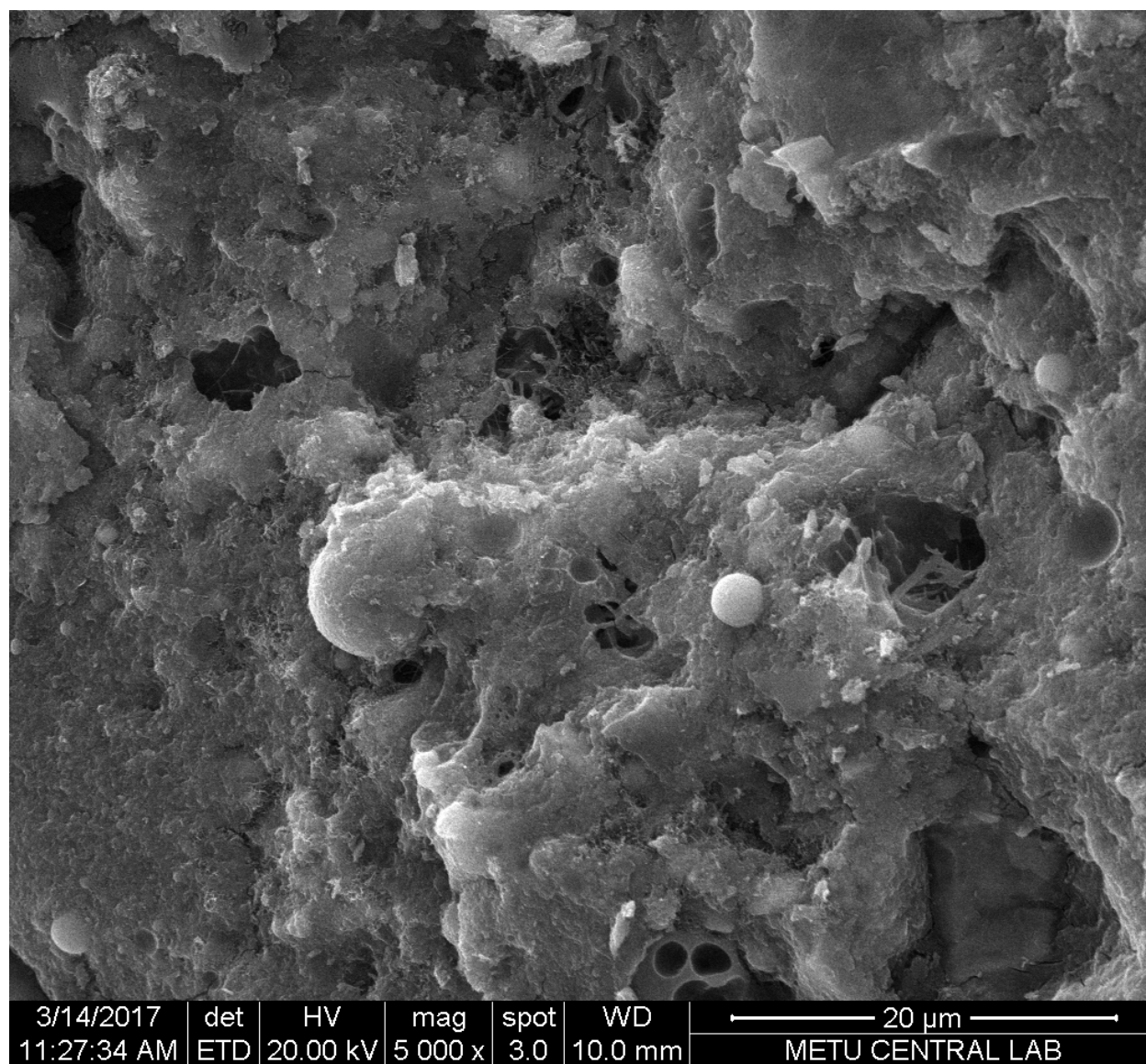


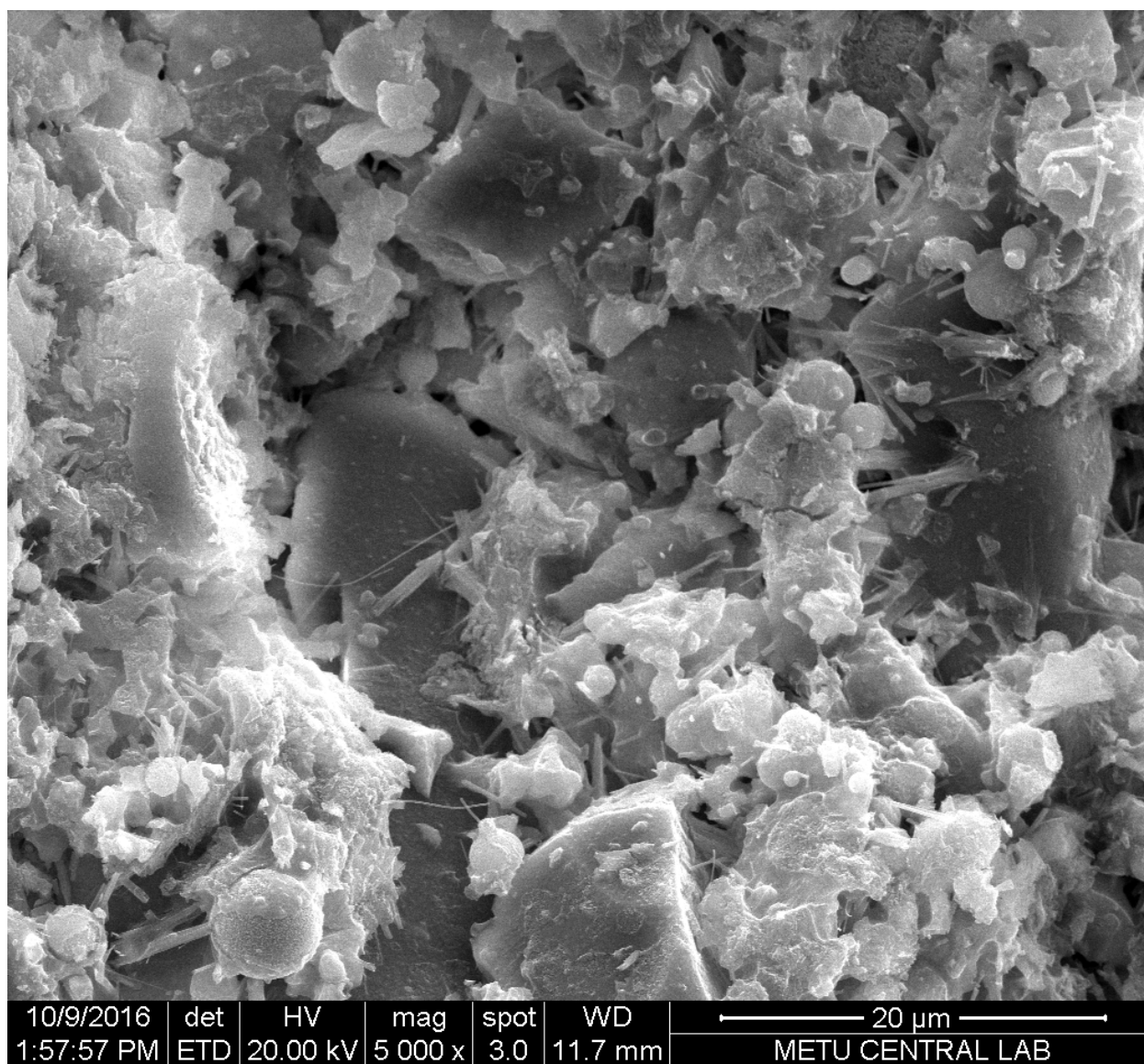




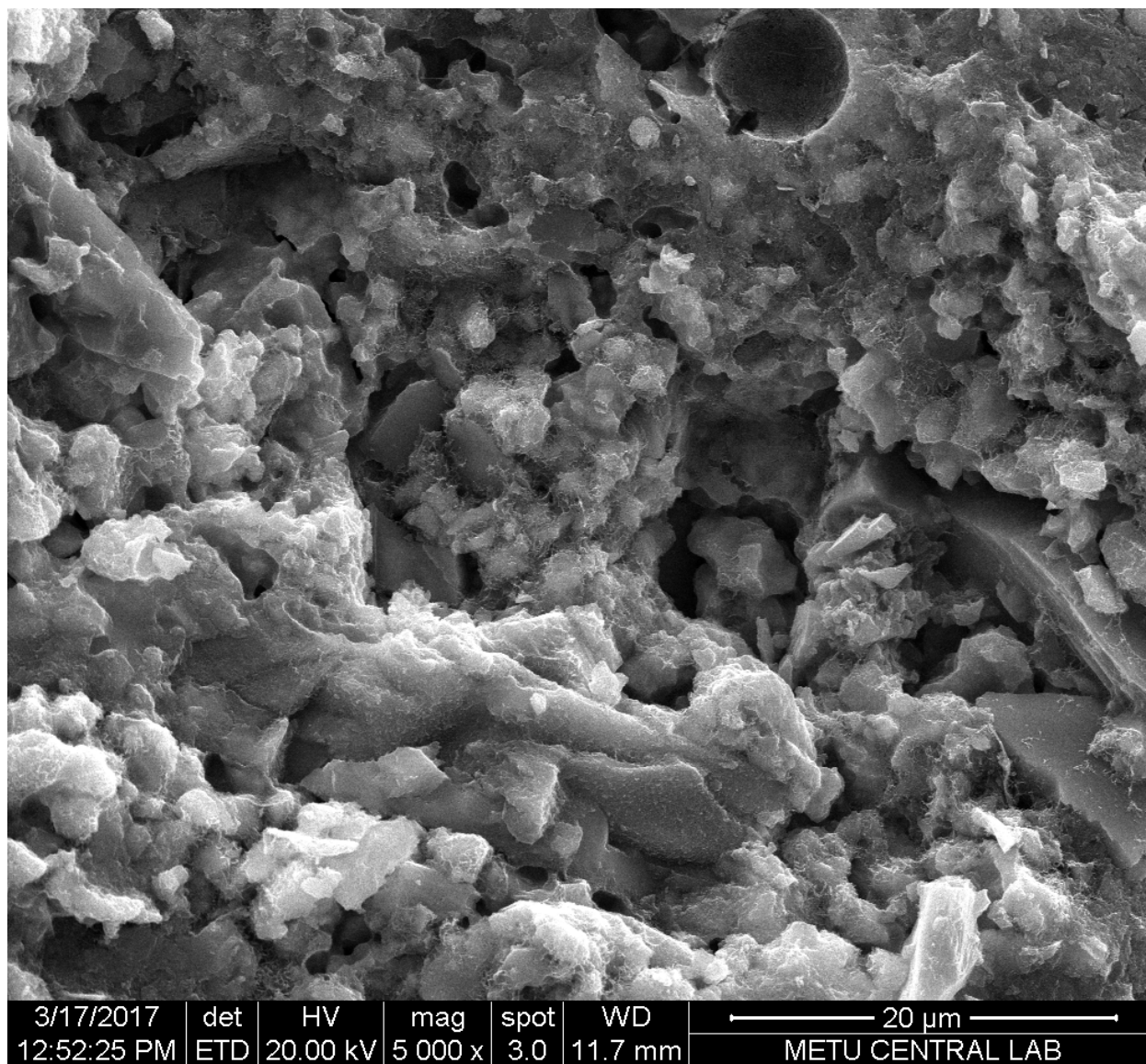




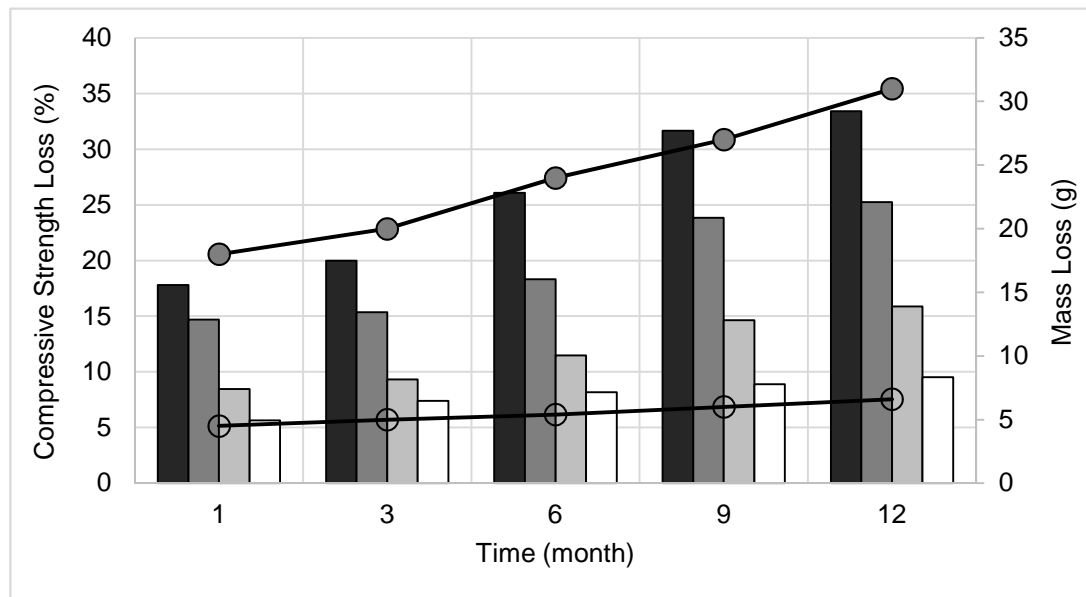












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Kind Regards,

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